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NAS SAUFLEY FIELD
5090.3a

FINAL SAMPLING AND ANALYSIS PLAN FOR SITE INVESTIGATION AT SITE 2 FIRE FIGHTER TRAINING AREA NAS SAUFLEY FIELD FL

9/1/2010

TETRA TECH

Comprehensive Long-term Environmental Action Navy

CONTRACT NUMBER N62470-08-D-1001

Rev. 0
September 2010

Final
Sampling and Analysis Plan
(Field Sampling Plan and Quality Assurance Project Plan)

Site Investigation
for
Site 2 Fire Fighter Training Area

Saufley Field
Pensacola, Florida

Contract Task Order JM30

September 2010

NAVFAC Naval Facilities Engineering Command
NAVFAC SOUTHEAST
NAS Jacksonville
Jacksonville, Florida 32212-0030

TETRA TECH NUS, Inc.

SAP Worksheet #1 -- Title and Approval Page
(UFP-QAPP Manual Section 2.1)

FINAL
SAMPLING AND ANALYSIS PLAN
(Field Sampling Plan and Quality Assurance Project Plan)
SEPTEMBER 2010

Site Investigation
Site 2 Fire Fighter Training Area
Saufley Field
Pensacola, Florida

Prepared for:
Naval Facilities Engineering Command Southeast
Naval Air Station Jacksonville Building 903
Jacksonville, Florida 32212-0030

Prepared by:
Tetra Tech NUS, Inc.
661 Andersen Drive
Foster Plaza 7
Pittsburgh, Pennsylvania 15220-2745

Prepared under:
Comprehensive Long-term Environmental Action Navy
CLEAN Contract Number N62470-08-D-1001
Contract Task Order JM30

SAP Worksheet #1 -- Approval Page
(UFP-QAPP Manual Section 2.1)

Document Title: Sampling and Analysis Plan (Field Sampling Plan and Quality Assurance Project Plan), June 2010, Site Investigation, Site 2 Fire Fighter Training Area, Saufley Field, Pensacola, Florida

Lead Organization: Naval Facilities Engineering Command Southeast

Preparer's Name and Organizational Affiliation: Yarissa Martínez, Tetra Tech NUS, Inc.

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(850) 385-9899

Preparation Date (Day/Month/Year): September 16, 2010

Investigative Organization's Project Manager:

Frank Lesesne 16 Sept 10
Signature/Date
Frank Lesesne
Tetra Tech NUS, Inc.

Investigative Organization's Project Quality Assurance Manager:

T. Johnston 16 Sep 10
Signature/Date
Tom Johnston, PhD
Tetra Tech NUS, Inc.

Lead Organization's Project Manager:

REED.SARAH.M.1239517036 Digitally signed by REED.SARAH.M.1239517036
DN: c=US, o=U.S. Government, ou=DoD, ou=PKI, ou=USN, cn=REED.SARAH.M.1239517036
Date: 2010.09.21 15:04:46 -04'00'

Signature/Date
Sarah Reed
Naval Facilities Engineering Command Southeast

Lead Organization Quality Assurance Officer:

BOWERS.KENNETH.A.1230092474 Digitally signed by BOWERS.KENNETH.A.1230092474
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Date: 2010.09.21 13:05:54 -04'00'

Signature/Date
Navy Chemist/Quality Assurance Officer
Naval Facilities Engineering Command Atlantic

Approval Signature:

Signature/Date
David Grabka
Florida Department of Environmental Protection

EXECUTIVE SUMMARY

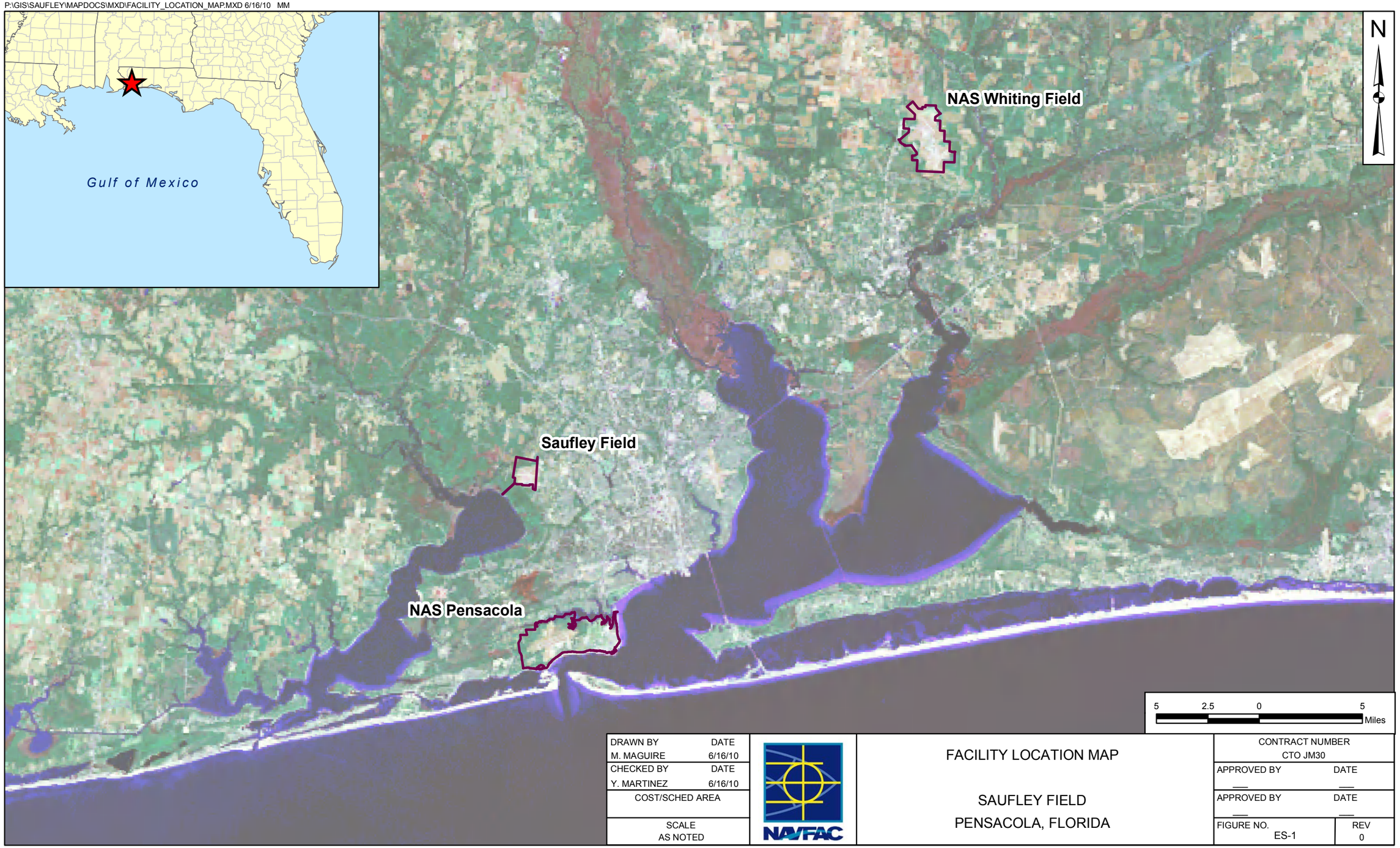
Tetra Tech NUS, Inc. has prepared this Uniform Federal Policy Sampling and Analysis Plan (UFP-SAP) under the Comprehensive Long-term Environmental Action Navy Contract Number N62470-08-D-1001, Contract Task Order JM30. This plan was prepared for surface soil, subsurface soil, and groundwater sampling events associated with completion of a Site Investigation (SI) for Site 2, Fire Fighter Training Area, at Saufley Field located in Pensacola, Florida. Figure ES-1 presents a Facility Location Map depicting the location of Saufley Field, and Figure ES-2 presents the location of the Site 2, Fire Fighter Training Area.

Saufley Field is located in Escambia County, between Interstate 10 and Perdido Bay, approximately 5 miles northwest of Pensacola, Florida, in the northwestern coastal section of the Florida panhandle. The installation's main complex currently encompasses approximately 866 acres and includes a number of support buildings, a federal prison located south of the airfield, four airstrips, and undeveloped lands. The area currently occupied by Saufley Field included farms and woodlands before it was purchased by the Navy in the 1930s.

According to the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 process, the SI follows a Preliminary Assessment. The primary objective of an SI is to determine whether further response actions or remedial investigation are appropriate for a site. During the SI, background information provided in any initial assessments is considered and supplemental site-specific environmental data are collected to further characterize the nature and extent of any present contamination, if any.

This UFP-SAP was generated for and complies with applicable United States Department of the Navy, United States Environmental Protection Agency (USEPA), and Florida Department of Environmental Protection requirements, regulations, guidance, and technical standards. This includes the Department of Defense, Department of Energy, and USEPA Interagency Data Quality Task Force (IDQTF) environmental requirements regarding federal facilities. To comply with IDQTF requirements, this UFP-SAP is presented in the format of 37 standard worksheets specified in the Uniform Federal Policy for Quality Assurance Project Plans guidance documents (USEPA, 2005).

P:\GIS\SAUFLEY\MAPDOCS\MXD\FACILITY_LOCATION_MAP.MXD 6/16/10 MM



DRAWN BY M. MAGUIRE DATE 6/16/10		FACILITY LOCATION MAP	CONTRACT NUMBER CTO JM30	
CHECKED BY Y. MARTINEZ DATE 6/16/10			APPROVED BY ___ DATE ___	
COST/SCHED AREA		SAUFLEY FIELD	APPROVED BY ___ DATE ___	
SCALE AS NOTED	NAVFAC	PENSACOLA, FLORIDA	FIGURE NO. ES-1	REV 0

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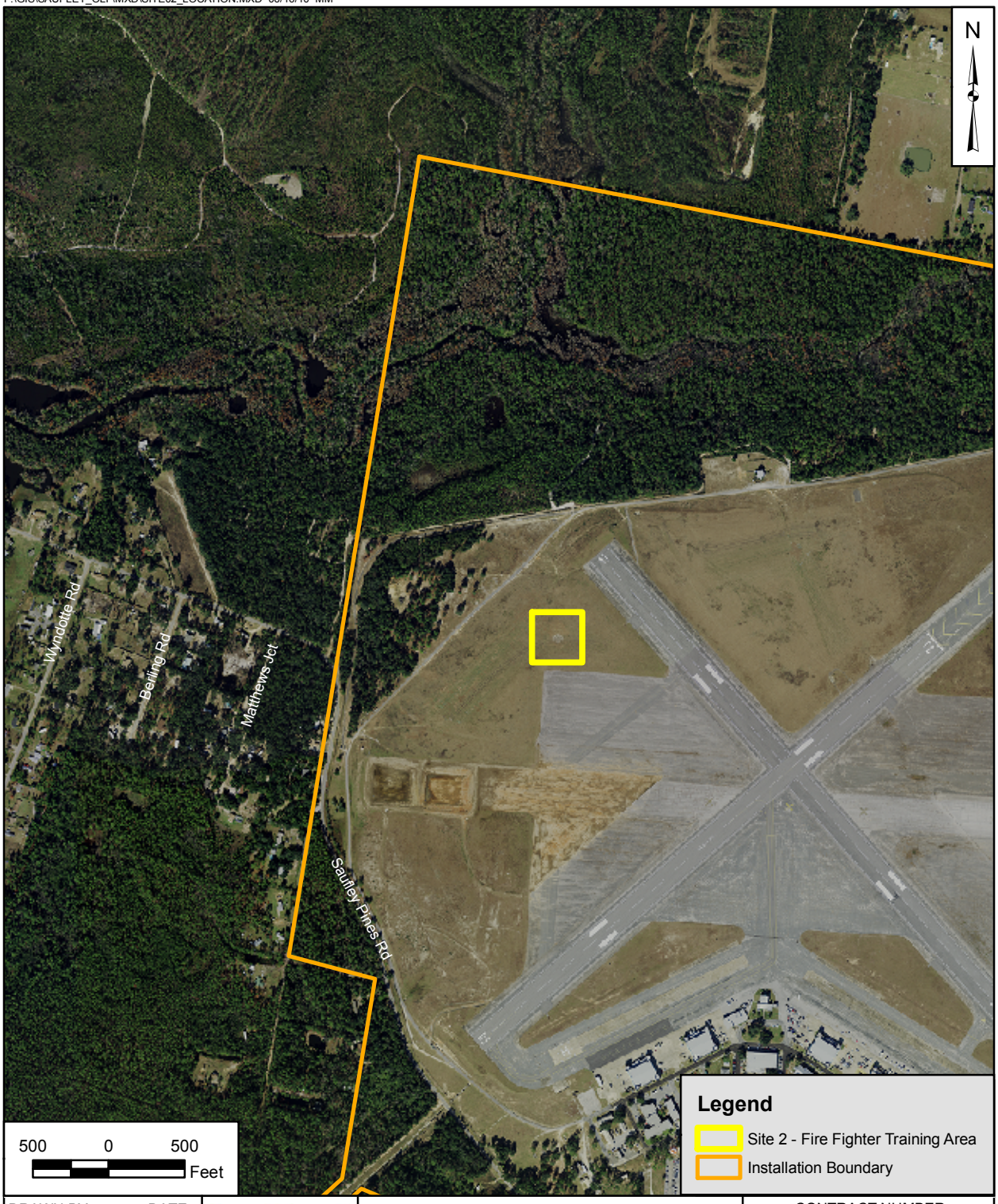


DRAWN BY M. MAGUIRE	DATE 06/16/10	SITE LOCATION SAUFLEY FIELD PENSACOLA, FLORIDA	CONTRACT NUMBER CTO JM30
CHECKED BY Y. MARTINEZ	DATE 06/16/10		APPROVED BY ___ DATE ___
COST/SCHED AREA			APPROVED BY ___ DATE ___
SCALE AS NOTED			FIGURE NO. ES-2 REV 0

SAP Worksheets

NUMBER **PAGE**

APPENDICES

LIST OF FIGURES

NUMBER **PAGE**

ACRONYMS AND ABBREVIATIONS

°C	Degree Celsius
%D	Percent Difference or Percent Drift
%R	Percent Recovery
%RSD	Percent Relative Standard Deviation
µg/L	Microgram per Liter
AES	Atomic Emission Spectroscopy
BFB	Bromofluorobenzene
bgs	Below Ground Surface
BHC	Benzene Hexachloride
CAS	Chemical Abstract Service
CCB	Continuing Calibration Blank
CCC	Calibration Check Compound
CCV	Continuing Calibration Verification
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act of 1980
CLEAN	Comprehensive Long-term Environmental Action Navy
CLLE	Continuous Liquid to Liquid Extraction
CSM	Conceptual Site Model
CTL	Cleanup Target Level
CTO	Contract Task Order
CVAA	Cold Vapor Atomic Absorption
cVOC	Chlorinated Volatile Organic Compound
DDD	Dichlorodiphenyldichloroethane
DDE	Dichlorodiphenyldichloroethylene
DDT	Dichlorodiphenyltrichloroethane
DFTPP	Decafluorotriphenylphosphine
DL	Detection Limit
DO	Dissolved Oxygen
DoD	Department of Defense
DPT	Direct Push Technology
DQI	Data Quality Indicator
DQO	Data Quality Objective
DVM	Data Validation Manager
ECD	Electron Capture Detector
EDD	Electronic Data Deliverable
EE/CA	Engineering Evaluation/Cost Analysis

ACRONYMS AND ABBREVIATIONS (continued)

ELAP	Environmental Laboratory Accreditation Program
Ext.	Extension
F.A.C.	Florida Administrative Code
FDEP	Florida Department of Environmental Protection
FID	Flame Ionization Detector
FL-PRO	Florida Petroleum Residual Organic Matter
FOL	Field Operations Leader
FTMR	Field Task Modification Request
g	Gram
GC/ECD	Gas Chromatography/Electron Capture Detector
GC/MS	Gas Chromatography/Mass Spectrometer
GCTL	Groundwater Cleanup Target Level
HASP	Health and Safety Plan
HCl	Hydrochloric Acid
HSM	Health and Safety Manager
ICAL	Initial Calibration
ICB	Initial Calibration Blank
ICP	Inductively Coupled Plasma
ICS	Interference Check Standard
ICV	Initial Calibration Verification
IDQTF	Interagency Data Quality Task Force
IDW	Investigation Derived Waste
IRP	Installation Restoration Program
IS	Internal Standard
Katahdin	Katahdin Analytical Services
KB Labs	KB Labs, Inc.
L	Liter
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LIF	Laser Induced Fluorescence
LOD	Limit of Detection
LOQ	Limit of Quantitation
mg/kg	Milligram per Kilogram
MIP	Membrane Interface Probe
mL	Milliliter

ACRONYMS AND ABBREVIATIONS (continued)

MPC	Measurement Performance Criterion
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NA	Not Applicable
NAS	Naval Air Station
NAVFAC	Naval Facilities Engineering Command
Navy	United States Department of the Navy
NEESA	Naval Energy and Environmental Support Activity
NELAP	National Environmental Laboratory Accreditation Program
NETPDTC	Naval Education and Training Professional Development and Technology Center
NFA	No Further Action
NTU	Nephelometric Turbidity Unit
OVA	Organic Vapor Analyzer
oz	Ounce
PA	Preliminary Assessment
PAH	Polycyclic Aromatic Hydrocarbon
PAL	Project Action Limit
PCB	Polychlorinated Biphenyl
PM	Project Manager
PoC	Point of Contact
PPE	Personal Protective Equipment
PQL	Practical Quantitation Limit
PQLG	Practical Quantitation Limit Goal
PT	Proficiency Testing (previously known as performance evaluation sample)
PWC	Public Works Center
QA	Quality Assurance
QAM	Quality Assurance Manager
QAO	Quality Assurance Officer
QC	Quality Control
r	Linear Regression Correlation Coefficient
r^2	Coefficient of Determination
RAP	Remedial Action Plan
RF	Response Factor
RI	Remedial Investigation
RPD	Relative Percent Difference

ACRONYMS AND ABBREVIATIONS (continued)

RPM	Remedial Project Manager
RRT	Relative Retention Time
RSL	Regional Screening Level
RT	Retention Time
SAP	Sampling and Analysis Plan
SCTL	Soil Cleanup Target Level
SD	Serial Dilution
SE	Southeast
SI	Site Investigation
SIM	Selected Ion Monitoring
SOP	Standard Operating Procedure
SPCC	System Performance Check Compound
SPP	Systematic Project Planning
SQL	Structured Query Language
SSO	Site Safety Officer
SVOC	Semivolatile Organic Compound
TBD	To Be Determined
TCL	Target Compound List
TCLP	Toxicity Characteristic Leaching Procedure
Tetra Tech	Tetra Tech NUS, Inc.
TRPH	Total Recoverable Petroleum Hydrocarbons
UFP-QAPP	Uniform Federal Policy for Quality Assurance Project Plan
UFP-SAP	Uniform Federal Policy Sampling and Analysis Plan
USEPA	United States Environmental Protection Agency
VOC	Volatile Organic Compound

SAP Worksheet #2 -- SAP Identifying Information
(UFP-QAPP Manual Section 2.2.4)

Site Name/Number:	Saufley Field, Pensacola, Florida
Operable Units:	Site 2, Fire Fighter Training Area
Contractor Name:	Tetra Tech NUS, Inc. (Tetra Tech)
Contract Number:	N62470-08-D-1001
Contract Title:	Comprehensive Long-term Environmental Action Navy (CLEAN)
Work Assignment Number:	Contract Task Order (CTO) JM30

1. This Sampling and Analysis Plan (SAP) was prepared in accordance with the requirements of the United States Environmental Protection Agency (USEPA) *Uniform Federal Policy for Quality Assurance Project Plans* (UFP-QAPP) (USEPA, 2005) and USEPA *Guidance for Quality Assurance Project Plans* (USEPA, 2002b).

2. Identify regulatory program: Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA).

3. This SAP is a project-specific SAP.

4. List dates of scoping sessions that were held:

Scoping Session	Date
Data Quality Objectives (DQOs) Meeting	May 10, 2010

5. List dates and titles of any SAP documents written for previous site work that are relevant to the current investigation:

Title	Date
Not Applicable (NA) – This is the initial site investigation (SI) for Site 2	

6. List organizational partners (stakeholders) and connection with lead organization:

   Florida Department of Environmental Protection (FDEP) (regulatory stakeholder)

   Naval Air Station (NAS) Pensacola (property owner)

7. Lead organization: Naval Facilities Engineering Command (NAVFAC) Southeast (SE)

8. If any required SAP elements or required information are not applicable to the project or are provided elsewhere, then note the omitted SAP elements and provide an explanation for their exclusion below:

   NA as there are no exclusions.

SAP Worksheet #3 -- Distribution List
(UFP-QAPP Manual Section 2.3.1)

Name of SAP Recipients	Title/Role	Organization	Telephone Number	E-Mail Address or Mailing Address
Bill Gates	NAVFAC SE Remedial Project Manager (RPM) – Manages Project Activities for the United States Department of the Navy (Navy)	NAVFAC SE Integrated Product Team South Central Code OPZE3 Building 903 Jacksonville, FL 32212-0030	(843) 763-5177	william.gates@navy.mil
Greg Campbell	Installation Restoration Program (IRP) Manager – NAS Pensacola Point of Contact (PoC)	NAS Pensacola Public Works Center (PWC) 310 John Tower Road Pensacola, FL 32508-5000	(850) 452-3131 Extension (Ext.) 3007	gregory.campbell@navy.mil
To Be Determined (TBD)	NAVFAC Quality Assurance (QA) Officer (QAO) – Navy Chemist	TBD	TBD	TBD
TBD	Head of Reference Desk (Saufley Field Administrative Record)	TBD	TBD	TBD
David Grabka	FDEP RPM – Provides Regulator Input	FDEP 2600 Blair Stone Road, MS 4535 Tallahassee, FL 32399-2400	(850) 245-8997	david.grabka@dep.state.fl.us
John Trepanowski (copy of cover letter only)	Tetra Tech Program Manager – Manages Navy Initiatives	Tetra Tech 234 Mall Boulevard Suite 260 King of Prussia, PA 19406	(610) 382-1532	john.trepanowski@tetratech.com
Garth Glenn (copy of cover letter only)	Tetra Tech Deputy Program Manager – Manages Program Activities	Tetra Tech 5700 Lake Wright Drive Suite 309 Norfolk, VA 23502	(757) 461-3926	garth.glenn@tetratech.com
Frank Lesesne	Tetra Tech Project Manager (PM) – Manages Project Activities	Tetra Tech 1558 Village Square Boulevard Suite 2 Tallahassee, FL 32309	(850) 385-9899 Ext. 1353	frank.lesesne@tetratech.com
TBD	Tetra Tech Field Operations Leader (FOL) /Site Safety Officer (SSO) – Manages Field Operation and Site Safety Issues	Tetra Tech TBD	TBD	TBD@tetratech.com

Name of SAP Recipients	Title/Role	Organization	Telephone Number	E-Mail Address or Mailing Address
Tom Johnston, PhD (electronic copy only)	Tetra Tech QA Manager (QAM) – Manages Corporate QA Program and Implementation	Tetra Tech 661 Andersen Drive Foster Plaza 7 Pittsburgh, PA 15220	(412) 921-8615	tom.johnston@tetratech.com
Matt Soltis (Health and Safety Plan [HASP] only)	Tetra Tech Health and Safety Manager (HSM) – Manages Corporate Health and Safety Program	Tetra Tech 661 Andersen Drive Foster Plaza 7 Pittsburgh, PA 15220	(412) 921-8912	matt.soltis@tetratech.com
Mark Traxler (electronic copy only)	Tetra Tech Project Chemist – Provides Coordination with Laboratory	Tetra Tech 234 Mall Blvd. Ste. 260 King Of Prussia, PA 19406	(610) 382-1171	mark.traxler@tetratech.com
Joseph Samchuck (electronic copy only)	Tetra Tech Data Validation Manager (DVM) – Manages Data Validation	Tetra Tech 661 Andersen Drive Foster Plaza 7 Pittsburgh, PA 15220	(412) 921-8510	joseph.samchuck@tetratech.com
Lee Leck (electronic copy only)	Tetra Tech Data Manager – Manages Databases	Tetra Tech 661 Andersen Drive Foster Plaza 7 Pittsburgh, PA 15220	(412) 921-8856	lee.leck@tetratech.com
Kate Zaleski (electronic copy only)	Laboratory PM – Representative for Laboratory and Analytical Issues	Katahdin Analytical Services, Inc. (Katahdin) 600 Technology Way Scarborough, ME 04070	(207) 847-2400	kzaleski@katahdinlabs.com
Todd Romero (electronic copy only)	Laboratory PM – Representative for Laboratory and Analytical Issues	KB Labs, Inc. (KB Labs) 25132 SW 1st Ave Newberry, FL 32669	(352) 472-5830	toddr@kbmobilelabs.com
TBD (electronic copy only)	Well Installation Subcontractor PM – Provides Membrane Interface Probe (MIP)/Laser Induced Fluorescence (LIF) and Direct Push Technology (DPT) Drilling Services	TBD	TBD	TBD

Each person in this table will be responsible for distributing copies of this SAP to appropriate personnel within their organization. For example, the Tetra Tech PM will be responsible for distributing copies of this SAP to all Tetra Tech personnel listed in Worksheet #4 (Project Personnel Sign-Off Sheet).

SAP Worksheet #4 -- Project Personnel Sign-Off Sheet

(UFP-QAPP Manual Section 2.3.2)

Certification that project personnel have read the text will be obtained by one of the following methods as applicable:

1. In the case of regulatory agency personnel with oversight authority, approval letters or e-mails will constitute verification that applicable sections of the SAP have been reviewed. Copies of regulatory agency approval letters/e-mails will be retained in the project files as project records (see Worksheet #29).

2. E-mails will be sent to the listed Navy, Tetra Tech, and subcontractor project personnel whom will be requested to verify by e-mail that they have read the applicable SAP/sections and the date on which they were reviewed. Copies of the verification e-mail will be included in the project files (see Worksheet #29).

   A copy of the signed Worksheet #4 will be retained in the project files and identified as a project document in Worksheet #29.

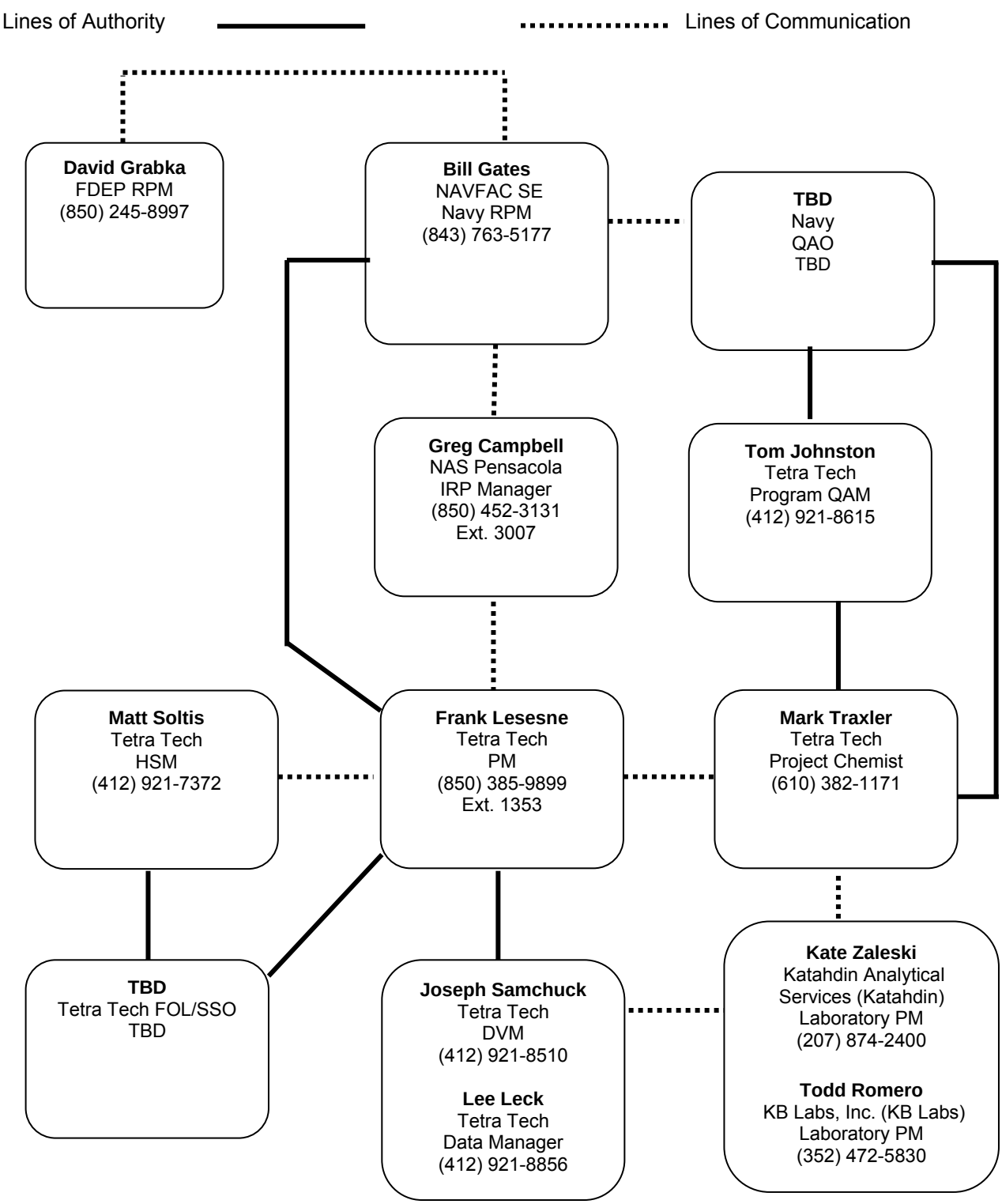
Key personnel will be instructed to read the SAP prior to attending an internal site-specific kick-off meeting for field activities. The Tetra Tech PM will track when the reviews have been completed, obtain signatures, and ensure that the completed sign-off sheet is included in the central project file.

Name [(1)]	Organization/Title/Role	Telephone Number	Signature/E-Mail Receipt	SAP Section Reviewed	Date SAP Read
Navy and Regulator Project Team Personnel					
Bill Gates	Navy/RPM – Manages Project Activities for the Navy	(843) 763-5177	See Worksheet #1 for signature	All	
Greg Campbell	Navy/IRP Manager – NAS Pensacola PoC	(850) 452-3131 Ext. 3007		All	
David Grabka	FDEP/RPM – Provides Regulator Input	(850) 245-8997	See Worksheet #1 for signature	All	

Name [1]	Organization/Title/Role	Telephone Number	Signature/E-Mail Receipt	SAP Section Reviewed	Date SAP Read
Tetra Tech Project Team Personnel					
Frank Lesesne	Tetra Tech/PM – Manages Project Activities	(850) 385-9899 Ext. 1353	See Worksheet #1 for signature	All	
TBD	Tetra Tech/FOL/SSO – Manages Field Operation and Site Safety Issues	TBD		All	
Tom Johnston	Tetra Tech/QAM – Manages NAVFAC SE Contract QA Program and Implementation	(412) 921-8615	See Worksheet #1 for signature	All	
Matt Soltis	Tetra Tech/HSM – Manages Corporate Health and Safety Program	(412) 921-8912	See HASP for signature	HASP	
Peggy Churchill	Tetra Tech/Environmental Scientist – Provides DQO and SAP Support	(321) 636-6470		All	
Mark Traxler	Tetra Tech/Project Chemist – Provides Coordination with Laboratory	(610) 382-1171		All	
Joseph Samchuck	Tetra Tech/DVM – Manages Data Validation	(412) 921-8510		Worksheets #12, #14, #15, #19, #20, #23-28, #30, and #34-37	
Lee Leck	Tetra Tech/Data Manager – Manages Databases	(412) 921-8856		Worksheets #12, #14, #15, #19, #20, #23-28, #30, and #34-37	
Subcontractor Personnel					
Kate Zaleski	Katahdin/Laboratory PM – Representative for Laboratory and Analytical Issues	(207) 874-2400		Worksheets #6, #12, #14, #15, #19, #23-28, #30, and #34-36	
Todd Romero	KB Labs/Laboratory PM – Representative for Laboratory and Analytical Issues	(352) 472-5830		Worksheets #6, #15, #19, #23, #24, #25, and #28	
TBD	TBD/Subcontractor PM – Driller for MIP/LIF, DPT, and Monitoring Well Installation	TBD		Worksheets # 6, #14, #17, and Figures	

Footnote: [1] - Persons listed on this worksheet will be responsible for distributing the SAP to the appropriate people within their organization.

SAP Worksheet #5 -- Project Organizational Chart
(UFP-QAPP Manual Section 2.4.1)



SAP Worksheet #6 -- Communication Pathways

(UFP-QAPP Manual Section 2.4.2)

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or E-Mail	Procedure
SAP amendments	Tetra Tech FOL/SSO Tetra Tech PM Navy RPM	TBD Frank Lesesne Bill Gates	TBD (850) 385-9899 Ext. 1353 (843) 763-5177	The Tetra Tech FOL will verbally inform the Tetra Tech PM within 24 hours of realizing a need for an amendment. The Tetra Tech PM will document the proposed changes via a Field Task Modification Request (FTMR) form within 5 days and send the Navy RPM a concurrence letter within 7 days of identifying the need for change. SAP amendments will be submitted by the Tetra Tech PM to the Navy RPM for review and approval. The Navy RPM will notify the regulators of changes to the SAP. The Tetra Tech PM will send scope changes to the Project Team via e-mail within 1 business day.
Schedule changes	Tetra Tech PM Navy RPM NAS Pensacola IRP Manager	Frank Lesesne Bill Gates Greg Campbell	(850) 385-9899 Ext. 1353 (843) 763-5177 (850) 452-3131 Ext. 3007	The Tetra Tech PM will verbally inform the Navy RPM and the NAS Pensacola IRP Manager on the day that schedule change is known and document via schedule impact letter within 1 business day of when impact is realized.
Field issues that require changes in scope or implementation of field work	Tetra Tech FOL/SSO Tetra Tech PM Navy RPM NAS Pensacola IRP Manager	TBD Frank Lesesne Bill Gates Greg Campbell	TBD (850) 385-9899 Ext. 1353 (843) 763-5177 (850)-452-3131 Ext. 3007	The Tetra Tech FOL will verbally inform the Tetra Tech PM on the day the issue is discovered. The Tetra Tech PM will inform the Navy RPM and the NAS Pensacola IRP Manager (verbally or by e-mail) of the issue within one day of the discovery. The Navy RPM will issue scope change (verbally or via e-mail), if warranted. The scope change is to be implemented before further work is executed. The Tetra Tech PM will also send a concurrence letter to the Navy RPM within 7 days, if project scope is affected. The Navy RPM will sign the letter within 5 days of receipt. The Tetra Tech PM will document the change(s) via an FTMR form within two days of identifying the need for change and will obtain required approvals within 5 days of initiating the form.

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or E-Mail	Procedure
Stop work recommendations (for example, to protect workers from unsafe conditions/situations or to prevent a degradation in quality of work) and initiate work upon corrective action	Tetra Tech FOL/SSO Tetra Tech PM Tetra Tech QAM Tetra Tech HSM Tetra Tech Project Chemist Navy RPM NAS Pensacola IRP Manager	TBD Frank Lesesne Tom Johnston Matt Soltis Mark Traxler Bill Gates Greg Campbell	TBD (850) 385-9899 Ext. 1353 (412) 921-8615 (412) 921-8912 (610) 382-1171 (843) 763-5177 (850) 452-3131 Ext. 3007	If Tetra Tech is the responsible party for a stop work command, the Tetra Tech FOL will inform onsite personnel, subcontractor(s), the NAS Pensacola IRP Manager, and the identified Project Team members within one hour (verbally or by e-mail). If a subcontractor is the responsible party, the subcontractor PM must inform the Tetra Tech FOL within 15 minutes, and the Tetra Tech FOL will then follow the procedure listed above.
Corrective action for field program	Tetra Tech QAM Tetra Tech PM Navy RPM	Tom Johnston Frank Lesesne Bill Gates	(412) 921-8615 (850) 385-9899 Ext. 1353 (843) 763-5177	The Tetra Tech QAM will notify the Tetra Tech PM verbally or by e-mail within 1 business day that the corrective action has been completed. The Tetra Tech PM will then notify the Navy RPM within 1 business day (verbally or by e-mail).
Field data quality issues	Tetra Tech FOL/SSO Tetra Tech PM	TBD Frank Lesesne	TBD (850) 385-9899 Ext. 1353	The Tetra Tech FOL will inform the Tetra Tech PM verbally or by e-mail on the same day that a field data quality issue is discovered.
Laboratory data quality issues	Katahdin Laboratory PM KB Lab Laboratory PM Tetra Tech Project Chemist Tetra Tech PM Navy RPM	Kate Zaleski Todd Romero Mark Traxler Frank Lesesne Bill Gates	(615) 345-1115 (352) 472-5830 (610) 382-1171 (850) 385-9899 Ext. 1353 (843) 763-5177	The Laboratory PM will notify (verbally or via e-mail) the Tetra Tech Project Chemist within one business day of when an issue related to laboratory data is discovered. The Tetra Tech Project Chemist will notify (verbally or via e-mail) the data validation staff and the Tetra Tech PM within 1 business day. The Tetra Tech PM will notify the Navy RPM (verbally or via e-mail) of significant data quality issues within 1 business day of resolution.

SAP Worksheet #7 -- Personnel Responsibilities and Qualifications Table

(UFP-QAPP Manual Section 2.4.3)

The personnel from Tetra Tech and the analytical laboratory(s) responsible for implementing the SAP are identified in the following table. Resumes are available upon request.

Name	Title/Role	Organizational Affiliation	Responsibilities
Bill Gates	RPM – Manages project activities for the Navy	NAVFAC SE	Oversees project implementation, including scoping, data review, and evaluation.
Greg Campbell	NAS Pensacola PoC/ IRP Manager – Manages daily site activities related to this project	NAVFAC SE NAS Pensacola	Oversees site activities and participates in scoping, data review, evaluation, and reviews the SAP.
Dave Grabka	RPM – Provides regulatory input	FDEP	Participates in scoping, data review, evaluation, and approves the SAP on behalf of FDEP.
Frank Lesesne	PM – Manages project on a daily basis	Tetra Tech	Oversees project, financial, schedule, and technical day-to-day management of the project.
TBD	FOL/SSO – Manages field operation and site safety issues	Tetra Tech	As the FOL, supervises, coordinates, and performs field sampling activities. As the SSO, is responsible for on-site project specific health and safety training and monitoring site conditions. Details of these responsibilities are presented in the site-specific HASP.
Tom Johnston	QAM – Oversees program and project QA activities	Tetra Tech	Reviews the SAP and ensures quality aspects of the CLEAN program are implemented, documented, and maintained.
Matt Soltis	HSM – Oversees health and safety activities	Tetra Tech	Oversees Tetra Tech CLEAN Program Health and Safety Program.
Mark Traxler	Project Chemist – Conducts data validation and reporting	Tetra Tech	Participates in project scoping, prepares laboratory scopes of work, and coordinates laboratory-related functions with laboratory. Oversees data quality reviews and QA of data validation deliverables.
Joseph Samchuck	DVM – Oversees data validation activities	Tetra Tech	Manages data validation activities within Tetra Tech, including ensuring QA of data validation deliverables, providing technical advice on data usability, and coordinating and maintaining the data validation review schedule.
Lee Leck	Data Manager – Manages databases	Tetra Tech	Manages Tetra Tech databases and ensures correct input of data.
Kate Zaleski Todd Romero	Laboratory PM – Manages project	Katahdin KB Labs	Coordinates analyses with laboratory chemists, ensures that scope of work is followed, provides QA of data packages, and communicates with Tetra Tech project staff.
TBD	Well Installation Subcontractor PM – Driller for MIP/LIF, DPT, and Monitoring Well Installation	TBD	Ensures that project specific requirements are communicated to field personnel.

Note: In some cases, one person may be designated responsibilities for more than one position. For example, the Tetra Tech FOL will be responsible for SSO duties. This action will be performed only as credentials, experience, and availability permits.

SAP Worksheet #8 -- Special Personnel Training Requirements

(UFP-QAPP Manual Section 2.4.4)

Each site worker performing sampling of hazardous materials will be required to have completed a 40-hour course (and annual 8-hour refresher, if applicable) in Health and Safety Training as described under Occupational Safety and Health Administration 29 Code of Federal Regulations 1910.120(b)(4). Safety requirements are addressed in greater detail in the site-specific Tetra Tech HASP.

SAP Worksheet #9 -- Project Scoping Session Participants Sheet

(UFP-QAPP Manual Section 2.5.1)

Project Name: Saufley Field Projected Date(s) of Sampling: Fall 2010 Project Manager: Frank Lesesne	Site Name: Site 2 Site Location: Saufley Field, Pensacola, Florida

Date of Session: May 10, 2010
Scoping Session Purpose: Develop DQOs with the Navy to support Uniform Federal Policy Sampling and Analysis Plan (UFP-SAP) development

Name	Title	Affiliation	Phone Number	E-mail Address	Project Role
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Gerry Walker	Base Coordinator	Tetra Tech	(850) 385-9899 Ext. 1362	gerry.walker@tetratech.com	Tetra Tech Base Coordinator
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9.1 SCOPING MEETINGS SUMMARY

Flammable fuels used for fire fighter training at Site 2 may have consisted of the following:

- Waste aviation gasoline
- Automotive fuels
- Kerosene and diesel fuels
- Hydraulic fluids
- Chlorinated solvents

Based on these materials, the analytical program for the fixed-base laboratory will consist of Target Compound List (TCL) volatile organic compounds (VOCs), TCL semivolatile organic compounds (SVOCs), TCL pesticides and TCL polychlorinated biphenyls (PCBs), Florida Used Oil Group metals (arsenic, cadmium, chromium, and lead), and total recoverable petroleum hydrocarbons (TRPH) following the Florida Petroleum Residual Organic Matter (FL-PRO) procedure. The list of target analytes is provided in Worksheet #15. If PCBs are detected in the Site 2 soils, a decision will be made after the SI has been completed to include or not include analysis for dioxins/furans during subsequent investigations.

The environmental media potentially affected by releases to the environment include the following:

- Surface Soil – Contaminants released to the environment may have adversely affected soils beneath and adjacent to the Fire Fighter Training Area.
- Subsurface Soil – Contaminants released to the surface soils may exceed their properties for leachability and migrate to subsurface soils as a result of fluids used to extinguish the practice fires and during precipitation events.
- Groundwater – Contaminants released to the subsurface soils may exceed their properties for leachability and migrate to groundwater as a result of fluids used to extinguish the practice fires and during precipitation events.

The Project Team decided that VOCs will not be analyzed in soil samples collected from the ground surface to a depth of 0.5 feet below ground surface (bgs), but will be analyzed in soil samples collected from 0.5 to 2 feet bgs.

The project will proceed as a petroleum site (per Chapter 62-770, Florida Administrative Code [F.A.C.]), if target analytes detected at the site are indicative of petroleum related constituents. If the detected target analytes are not indicative of only petroleum related constituents, the site will proceed on a path similar to a CERCLA site, or Chapter 62-780, F.A.C.

Soil and groundwater Project Action Limits (PALs), if the site proceeds on a petroleum path, include the following:

- FDEP Residential Direct Exposure Soil Cleanup Target Levels (SCTLs) under Chapter 62-777, F.A.C.
- FDEP Industrial Direct Exposure under Chapter 62-777, F.A.C.
- FDEP Leachability to Groundwater under Chapter 62-777, F.A.C.
- Groundwater Cleanup Target Levels (GCTLs) under Chapter 62-777, F.A.C.
- Primary and Secondary Standards under Chapter 62-550, F.A.C.

Soil and groundwater PALs to be added to those listed above in the screening process, if the site proceeds on a path similar to CERCLA or Chapter 62-780, F.A.C., include the following:

- USEPA Regions 3, 6, and 9 Regional Screening Levels (RSLs) for Chemical Contaminants at Superfund Sites, Residential Direct Contact (R-RSL) (USEPA, 2010).
- USEPA Regions 3, 6, and 9 RSLs for Chemical Contaminants at Superfund Sites for Tap Water (T-RSL) (USEPA, 2010).

Decision rules include the following:

- If soil and groundwater do not exceed their PALs, then No Further Action (NFA).
- If soil or groundwater exceed their PALs, then based on the petroleum or CERCLA path decision, conduct additional assessment and/or a remedial action plan under Chapter 62-770, F.A.C., or an remedial investigation (RI) and/or Engineering Evaluation/Cost Analysis (EE/CA).

Decision to conduct additional assessment and/or a remedial action plan or RI or EE/CA will be based on the following:

- Media affected (soil and/or groundwater)
- Horizontal and vertical extent of contamination in affected media
- Magnitude of contamination in affected media
- Cost of RI versus EE/CA

SAP Worksheet #10 -- Conceptual Site Model
(UFP-QAPP Manual Section 2.5.2)

10.1 INTRODUCTION

The general location of Saufley Field is shown on Figure ES-1. Saufley Field is located on the Florida panhandle approximately 5 miles northwest of Pensacola, Florida. The installation currently encompasses approximately 866 acres and includes four airstrips, of which two are active, and a number of small buildings that are located south of the airfield. The majority of Saufley Field is covered by paved runway surrounded by mowed, open grassy fields and infrastructure for tenant support. Approximately 200 of the 866 acres are undeveloped. South of the airstrips, the majority of the adjacent area is predominantly wooded and supports a wide variety of flora and fauna.

Saufley Field opened in 1940, commissioned as a Naval Auxiliary Air Station, and was re-designated a NAS in 1968. It was decommissioned in 1976 and designated as an outlying landing field and reactivated in 1979 as a Naval Education and Training Program Development Center and as an outlying field for NAS Whiting Field pilot training. In 1996, Saufley Field became the Naval Education and Training Professional Development and Technology Center (NETPDTC), a major shore command. As the host of Saufley Field, NETPDTC supports 10 major Department of Defense (DoD), as well as Navy, tenants and has a total base population in excess of 1,000. Saufley Field operates two active runways and has in excess of 34,425 square feet of hangar space.

In 2008, the Navy entered into negotiations to form an Enhanced Use Lease partnership with private industry. The objective of the Enhanced Use Lease program is to transform 104 acres of the property at Saufley Field into a diversified, multi-use business campus through the creative adaptation and reuse of two sites. Site 1 contains 85.5 acres with 60 buildings (including 4 hangers) encompassing 622,000 square feet of space, and Site 2 contains 18.7 acres that is currently used as a golf course. The total site also offers potential access to two 4,000 linear foot runways.

The subject of this UFP-SAP is Site 2, which known as the Fire Fighter Training Area (see Figure ES-2). The Fire Fighter Training Area is located about 400 feet southwest of Runway 13. The Fire Fighter Training Area is a 60-foot diameter concrete pad surrounded by soil. Site 2 is located in the northwestern portion of Saufley Field and is generally located at or in the immediate vicinity of latitude 30º 28’ 21” North and 87º 20’ 46” West. The site elevation is approximately 70 feet North American Vertical Datum.

10.2 PHYSICAL SITE DESCRIPTION

10.2.4 Land Use

Saufley Field is an active military facility. Saufley Field, originally built and subsequently developed further to support various military activities including pilot training, is now used primarily to train and educate Navy personnel and to house federal prisoners. NAS Whiting Field pilots use two of the airstrips for touch and go landing exercises.

Currently, the primary mission of this facility is tenant support, which includes an Enhanced Use Lease partnership with private industry. Additional missions include use as an emergency landing location. Land use at Saufley Field is considered to be military/industrial.

There are no known future land use/development restrictions identified for Saufley Field.

10.2.5 Access Controls/Restrictions

Saufley Field is surrounded by a perimeter security fence; however, a separate fence or other barrier is not provided for Site 2. Access to the installation is restricted to Navy and civilian personnel, authorized contractors, and visitors.

10.3 PREVIOUS ENVIRONMENTAL INSPECTIONS AND INVESTIGATIONS AND REGULATORY STATUS

The Preliminary Assessment (PA) conducted in May 1992 by the Navy Energy and Environmental Support Activity identified the Fire Fighter Training area based on its operational history described below. Environmental samples were not collected at that time. The PA recommended that soil and/or groundwater sample be collected at the site; however, environmental sampling has not been conducted.

10.4 SITE HISTORY

The Fire Fighter Training Area is located about 400 feet southwest of Runway 13 (see Figure ES-2). The training area is a 60-foot diameter circular concrete pad surrounded by soil. The exact details of fire fighting training drills are unknown; however, a typical burn likely consisted of burning between 300 and 1,000 gallons of flammable liquids per training exercise. A typical fire fighting training drill likely consisted of covering the concrete pad with a flammable material and igniting it. The fire would be put out,

reignited, and put out again. The last fire fighting training drill was conducted in 1977 (approximately 33 years ago).

The majority of flammable liquids burned in the concrete pad were likely waste aviation gasoline, but other flammable liquids such as kerosene, chlorinated solvents, diesel, hydraulic fluids, and automobile gas may have been burned. Some hydraulic fluids prior to 1972 containing PCBs may have been burned. It is also thought by the Project Team that pesticides may have been mixed with the fluids being burned because the carrier fluid for pesticides at that time was typically a hydrocarbon-based fluid and of the potential closure of the facility in the 1960s. Additionally, because waste fuels and fluids were used in the fire training activities, the metals that would be most likely present from the flammable liquids burned during the training drills are the Florida Used Oil Group including arsenic, cadmium, chromium, and lead.

The soil in the site area consists of Lakeland sand, which is very permeable; therefore, while most of the flammable liquids were burned off, some may have leached into the soil. The concrete pad is flat, but the surrounding area drains to the northwest. Surface water runoff from the general site area drains toward Eight Mile Creek (a branch of Eleven Mile Creek), which is located approximately 1,900 feet to the north-northwest of Site 2.

10.5 CONCEPTUAL SITE MODEL

A conceptual site model (CSM) that provides a plan view of the source area, stratigraphy, hydrogeology, and contamination migration pathways is provided in Figure 10-1. The environmental media potentially affected by releases to the environment include the following:

- Surface Soil – Contaminants released to the environment may have adversely affected soils beneath and adjacent to the Fire Fighter Training Area.
- Subsurface Soil – Contaminants released to the surface soils may exceed their properties for leachability and migrate to subsurface soils as a result of fluids used to extinguish the practice fires and during precipitation events.
- Groundwater – Contaminants released to the subsurface soils may exceed their properties for leachability and migrate to groundwater as a result of fluids used to extinguish the practice fires and during precipitation events.

The purpose of the investigation activities is to collect additional data to refine the CSM and prepare a SI report. The data collected for the SI report will be used to evaluate the nature and extent of chemicals detected in soil and groundwater samples and to determine if the project will proceed as a petroleum site under Chapter 62-770, F.A.C., if target analytes detected at the site are indicative of petroleum related

constituents. If the target analytes are not indicative of only petroleum related constituents, the site will proceed on a path similar to a CERCLA site or under Chapter 62-780, F.A.C. Additionally, if soil and groundwater do not exceed their PALs, then a NFA decision is warranted. If soil or groundwater exceed their PALs, then based on the petroleum or CERCLA path decision, conduct additional assessment and/or a remedial action plan under Chapter 62-770, F.A.C., or an RI and/or EE/CA under Chapter 62-780, F.A.C.

10.5.1 Geology and Hydrogeology

In the southern one-half of Escambia County, the sand and gravel aquifer and the upper limestone of the Floridan aquifer are separated by a thick section of relatively impermeable clay; however, in the northern one-half of Escambia County, the sand and gravel aquifer and the upper limestone of the Floridan aquifer are in contact with one another. The upper limestone of the Floridan aquifer is separated from the lower limestone by a thick clay bed (Musgrove et. al., 1965).

The sand and gravel aquifer is composed of sand with numerous lenses and layers of clay and gravel. The formation also contains lenses of hardpan where the sand has been cemented by iron oxide minerals. This aquifer lies at the surface throughout Escambia County. Logs of borings from various locations throughout Saufley Field show that the surficial sands extend from ground surface to a depth of at least 129 feet mean sea level, below which is a 15-feet thick marine clay, the continuity of which is uncertain. Underlying the clay is more sand with numerous clay lenses (Geraghty and Miller, Inc. 1986).

Water levels in the shallow aquifer range from 27 feet (near the southeastern perimeter of the facility) to approximately 50 feet bgs near the western edge of Site 4. The groundwater flow has historically been toward the Gulf of Mexico and Escambia and Perdido Rivers; however, groundwater flow can vary locally due to the effect of topography or surface water bodies. Also, the aquifer recharge is predominantly from local precipitation (Trapp, 1973).

The shallow saturated permeable beds in the sand and gravel aquifer contain groundwater under nonartesian conditions, while the deeper permeable beds contain groundwater under artesian pressure, where they are confined by lenses of clay and sandy clay (Naval Energy and Environmental Support Activity [NEESA], 1983).

Below the sand and gravel aquifer, the limestone layers comprise the regionally extensive Floridan aquifer, which in this area is divided into upper and lower units separated by the Bucatunna clay. The upper Floridan aquifer is an important source of water in areas east of Escambia County; however, in the

Pensacola area it is highly mineralized and not used as a water supply. The lower Floridan aquifer is also highly mineralized and is designated for use as an injection zone (Geraghty and Miller, Inc. 1986).

10.5.2 Nature and Extent of Contamination

Currently, assessment activities have not been conducted nor have environmental samples (soil and groundwater) been collected from Site 2.

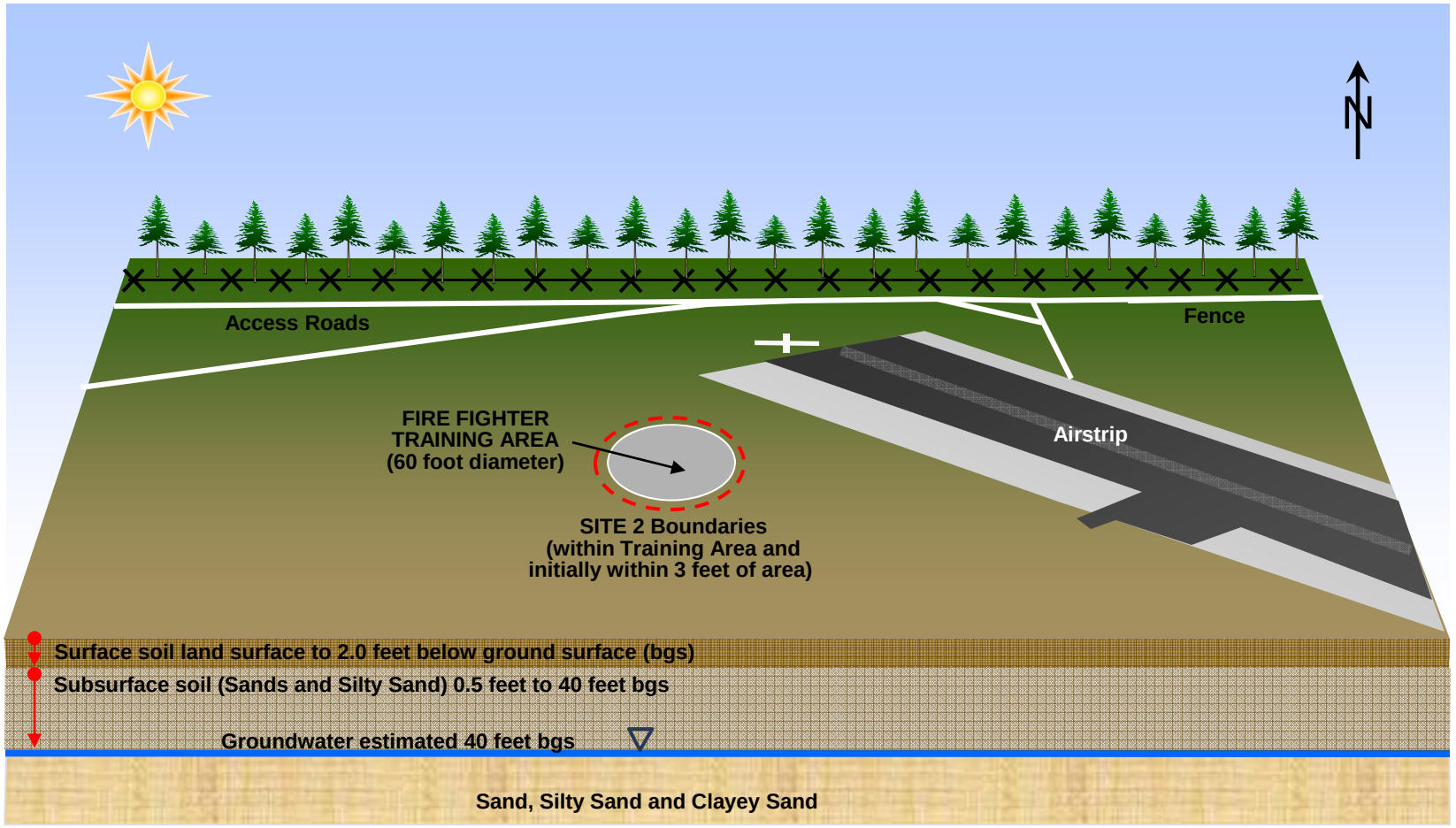
10.5.3 Migration Pathways

Contaminants released to the environment during fire fighting training activities may have adversely affected surface soils beneath and adjacent to the Fire Fighter Training Area and if they exceed their properties for leachability they could migrate to subsurface soils during precipitation events. Additionally, if the contaminants released to the subsurface soils exceed their properties for leachability they could migrate to groundwater during precipitation events.

10.5.4 Potential Receptors

Human receptors potentially include industrial workers, construction workers, maintenance workers, trespassers/recreational users, and hypothetical future residents. Because the current and future industrial use is not anticipated to change, maintenance workers and trespassers are considered to be the most likely receptors to contact contaminants that may be present in surface and subsurface soil at Site 2. The assumed exposure routes for contact with the surface and subsurface soil for the anticipated receptors include ingestion, dermal contact, and inhalation.

In 1994, the PWC potable water treatment system at Saufley Field included two active potable water wells. On May 9, 1994, a water sample from the potable water well PW04 effluent indicated benzene concentrations of 32 micrograms per liter (µg/L) exceeding the FDEP drinking water standard of 1 µg/L. Potable water well PW04 was taken off-line and was subsequently placed on quarterly sampling for one year for observation and corrective action to remove the contamination. In April 1996, potable water wells PW03 and PW04 were abandoned in place. Currently, the only source of potable water for Saufley Field is a well field located at the Naval Technical Training Center Corry Station, approximately 5.5 miles south of the installation. Therefore, groundwater from the site is not used as a water supply; however, an assumed exposure route to the hypothetical future resident for contact with groundwater exists.



DRAWN BY DATE: C. Pennington 5-10	NAVFAC	CONCEPTUAL SITE MODEL SAUFLEY FIELD, SITE 2 FIRE FIGHTER TRAINING AREA PENSACOLA, FLORIDA	CONTRACT NO.
CHECKED BY DATE: F. Lesesne 6-10			OWNER NO.
REVISED BY DATE			APPROVED BY DATE
SCALE: NOT TO SCALE			DRAWING NO.: FIGURE 10-1 / REV.

LEGEND

- Water Table
- Fence
- Site Boundary
- Site Boundary
- Limestone

SAP Worksheet #11 -- Project Quality Objectives/Systematic Planning Process Statements
(UFP-QAPP Manual Section 2.6.1)

The following text describes the development of the Project Quality Objectives using USEPA's DQO (System Planning) Process.

11.1 PROBLEM DEFINITION

Environmental data is required to refine the CSM and prepare an SI for Site 2. The SI will determine if surface and subsurface soil and groundwater within the boundary of Site 2 have been affected by potential contaminants that are related to the fire fighting training activities. The environmental data will be used to characterize the nature and extent of contaminants present in surface and subsurface soil and groundwater within the boundary of Site 2.

This data will be used to determine if the project will proceed as a petroleum site under Chapter 62-770, F.A.C., or if the site will proceed on a path similar to a CERCLA site under Chapter 62-780, F.A.C. The data will also be used to determine: if soil and groundwater do not exceed their PALs, then a NFA decision will be made; or, if soil or groundwater exceed their PALs, then determine if the exceedances are only petroleum-based, or if there are non-petroleum target analytes with exceedances to select a path forward, and based on the petroleum or CERCLA path decision, conduct additional assessment and/or a remedial action plan under Chapter 62-770, F.A.C., for a petroleum site, or an RI and/or EE/CA under Chapter 62-780, F.A.C., for a CERCLA site.

11.2 INFORMATION INPUTS

This sampling effort will utilize a Triad Approach to collect, evaluate, and prioritize data collection to evaluate the extent of contaminants in surface and subsurface soil and groundwater. A DPT rig with MIP/LIF will be used to collect data to provide information for real-time decision making for the collection of groundwater samples for field screening analysis of volatile organic vapors and for analysis of VOCs by KB Labs, a National Environmental Laboratory Accreditation Program (NELAP)-approved mobile laboratory. The MIP/LIF field screening results are real-time three dimensional semi-quantitative data that will be used to select samples (surface and subsurface soil and groundwater) for confirmation analysis by a fixed base laboratory and to select the locations of monitoring wells that will be used to collect groundwater samples.

The MIP/LIF three dimensional semi-quantitative data will be used to select soil samples that represent high, medium, low, and no responses by the MIP/LIF instrumentation. The confirmation soil samples selected by the field operation lead to represent the high, medium, and low MIP/LIF instrument response

will each consist of 30 percent of the 20 samples or 6 soil samples for each category and the confirmation soil samples selected to represent no responses by the MIP/LIF instrumentation will be 10 percent of the 20 samples or 2 soil samples.

The laboratory analytical results of the confirmation soil samples will be compared graphically to the MIP/LIF instrument response data to correlate, if possible, an approximate concentration of the target analytes to the MIP/LIF instrument response.

Groundwater samples will also be collected with the DPT at each of the 20 MIP/LIF DPT locations. The 20 DPT groundwater samples will be analyzed using a mobile laboratory to initially characterize whether or not a release to groundwater is present and its potential horizontal extent.

Replicates of the DPT groundwater samples will consist of a minimum of 5 percent (1 groundwater sample) up to a maximum of 5 of the 20 groundwater samples screened for VOCs by the on-site laboratory. The replicates will be analyzed off-site by Katahdin for definitive VOCs analysis. At a minimum, the replicates submitted for fixed-base laboratory analysis will represent low, medium, and high concentrations of the target analytes (e.g., low would be a concentrations at or below the detection limit for the target analytes detected by the mobile laboratory, medium would be at the approximate cleanup target levels (CTLs) for the target analytes detected by the mobile laboratory, and high would be at concentrations that exceeds the CTLs for the detected target analytes by the mobile laboratory.

The Project Team will use the DPT groundwater data, along with the results of the MIP/LIF data, to select locations to install up to 10 permanent shallow monitoring wells and three permanent deep monitoring wells to characterize the horizontal extent of a plume, should one exist. The monitoring well locations will be selected to represent hydraulic upgradient, side gradient, and downgradient locations that are similar to the horizontal distribution of the plume, if present, found at the 20 MIP/LIF DPT locations.

The confirmation soil sample analysis and groundwater samples collected from the monitoring wells will be analyzed for VOCs, SVOCs, pesticides, PCBs, Florida Waste Oil metals (arsenic, cadmium, chromium, and lead), and TRPH (using FL-PRO) by Katahdin, a DoD Environmental Laboratory Accreditation Program (ELAP) accredited and NELAP-approved fixed-base laboratory. The results of the environmental sampling of surface and subsurface soil and groundwater will be evaluated to establish the boundaries of Site 2 and to collect pre-design soil samples for the Site 2 EE/CA, if needed. Based on the previous site use, it is anticipated that the pre-design soil samples will be analyzed by Katahdin for a subset of the target analytes that are detected at Site 2 based on the findings for the laboratory analysis of the confirmatory soil samples. The subset of the target analytes for the pre-design soil sampling event will be selected by the Project Team after review the analytical results of the confirmatory soil samples.

The following physical and chemical data will be collected during this investigation:

1. MIP results: The MIP is a screening tool with semi-quantitative capabilities acting as an interface between contaminants (chlorinated volatile organic compounds [cVOCs]) in the subsurface and gas phase detectors at the surface. MIP acquisition software logs detector signal with depth. The detectors to be utilized include an electron capture detector (ECD) and a flame ionization detector (FID). The ECD is designed for sensitivity to cVOCs and other electronegative organic compounds, and the FID is a general detector designed for sensitivity to all combustible hydrocarbons. The ECD and FID will be used for VOCs field analysis.

2. LIF: The fiber optic-based LIF sensor system is light at a specific wavelength generated from a laser that is passed down a fiber optic cable to a sapphire window in the tip of the rod string as it is advanced into the subsurface. The laser light excites two or three ring aromatic compounds, or polycyclic aromatic hydrocarbons (PAHs), in the soil adjacent to the sapphire window causing them to fluoresce. The relative response of the sensor depends on the specific analyte being measured because of the varying ratios of PAHs in each hydrocarbon mixture. The induced fluorescence from the PAHs is returned over a second fiber to the surface, where it is quantified using a detector system. The peak wavelength and intensity provide information about the type of petroleum product.

3. Chemical Data: Surface and subsurface soil and groundwater samples will be analyzed by Katahdin for the select list of target analytes that are presented in Worksheet #15. Groundwater samples will also be analyzed by a mobile laboratory for only VOCs. The sampling methods that will be utilized are presented in Worksheet #18, and the analytical methods are presented in Worksheet #19.

4. Field Parameters: Field investigation parameters for groundwater will include dissolved oxygen (DO), oxidation-reduction potential, pH, conductivity, temperature, and turbidity. These data will be collected in the field. The relevant Standard Operating Procedures (SOPs) are presented in Worksheet #21.

5. Groundwater Level Measurements: Synoptic groundwater levels will be measured in each monitoring well to determine the groundwater flow direction. The sampling methods are presented in Worksheet #18.

6. PALs: Concentrations of target analytes will be compared against PALs. The PALs for this SI are derived from the following criteria for each media of concern:

Soil

If the site is evaluated and it is determined by the Project Team to be a petroleum site under Chapter 62-770, F.A.C., then the PALs will consist of the following:

- SCTLs for Chapter 62-777, F.A.C., Table II (Soils) – residential only, not leachability.
- The laboratory Practical Quantitation Limit (PQL) should be used if it is less stringent than the CTL according to Chapter 62-780.680(2)(b)2.a.(III), F.A.C. The PQL, as defined by the FDEP, is the lowest concentration that a laboratory can accurately report on a chemical. The Project Team has agreed to replace the PALs with the laboratory Limit of Quantitation (LOQ) for decision making purposes, as suggested in "Guidance for the Selection of Analytical Methods for the Evaluation of Practical Quantitation Limits" (FDEP, 2004).

If the site is evaluated and it is determined by the Project Team to proceed on a path similar to a CERCAL site under Chapter 62-780, F.A.C., then the PALs will also consist of the following:

- Derived Alternative CTLs per Chapter 62-780(5), F.A.C.
- Apportioned SCTLs in accordance with Chapter 62-780(2)(b)1.2.(V), F.A.C.
- 95% Upper Confidence Limit approach in accordance with Chapter 62-780.680(2)(b)1.2.(II), F.A.C.
- USEPA Regions 3, 6, and 9 RSLs for Chemical Contaminants at Superfund Sites – Residential Soil Values (R-RSLs) (USEPA, 2010).
- The laboratory PQL should be used if it is less stringent than the CTL according to Chapter 62-780(2)(b)2.a.(III), F.A.C.

Groundwater

If the site is evaluated and it is determined by the Project Team to be a petroleum site under Chapter 62-770, F.A.C., then the PALs will consist of the following:

- FDEP GCTLs per Chapter 62-777, F.A.C., Table 1 (Groundwater).
- Florida Drinking Water Standards, Chapter 62-550.310, F.A.C.
- Chapter 62-780, F.A.C., definition for low yield, poor quality aquifers.
- The laboratory PQL should be used if it is less stringent than the CTL according to Chapter 62-780(1)(c), F.A.C. However, the Project Team has agreed to replace the PALs with the laboratory LOQs for decision making purposes, as suggested in "Guidance for the Selection of Analytical Methods for the Evaluation of Practical Quantitation Limits" (FDEP, 2004).

If the site is evaluated and it is determined by the Project Team to proceed on a path similar to a CERCLA site under Chapter 62-780, F.A.C., then the PALs will also consist of the following:

- USEPA Maximum Contaminant Levels.
- USEPA Regions 3, 6, and 9 RSLs for Chemical Contaminants at Superfund Sites – Tap Water Values (T-RSLs) (USEPA, 2010).

11.3 STUDY AREA BOUNDARIES

The Site 2 horizontal boundary will initially consist of the Fire Fighter Training Area and within 3 feet of the Fire Fighter Training Area. The vertical boundary will initially consist of the following.

- Surface Soil – 0 to 2 feet bgs
- Subsurface Soil – 2 to 40 feet bgs or the top of the water table, whichever comes first.

Soil samples will be collected until MIP, LIF, or positive organic vapor analyzer (OVA) (until OVA FID results are no longer detected or until groundwater is intercepted). Groundwater is estimated to be 40 feet bgs. Groundwater samples will be collected from a screened interval that is placed from the water table to 5 feet below the water table for analysis by a mobile laboratory. If the soil and groundwater screening criteria are exceeded, the Site 2 boundary will be expanded by stepping out approximately 20 feet no more than three times.

11.4 ANALYTIC APPROACH

Separate decision rules have been created for the regulatory path and for the evaluation of the soil and groundwater migration pathways being investigated. The decision rules are presented below.

11.4.1 Regulatory Path Decision Rule

If target analytes detected in exceedance of the applicable PALs are indicative of only petroleum related constituents as identified in Tables A and B of Chapter 62-770, F.A.C., then the project will proceed as a petroleum site under Chapter 62-770, F.A.C.

If the target analytes detected in exceedance of the applicable PALs are not indicative of only petroleum related constituents, then the site will proceed on a path similar to a CERCLA site under Chapter 62-780, F.A.C.

11.4.2 Surface Soil Decision Rule

Individual surface and subsurface soil concentrations will be compared to soil PALs.

- If surface and/or subsurface soil target analyte concentration does not exceed their PALs for target analytes, collected below the Fire Fighter Training Pit or the 3-foot step out, then NFA for soils will be recommended.
- If any surface and/or subsurface soil target analyte concentration exceeds the PAL for that target analyte, additional data will be collected via step out samples.
- If any surface and/or subsurface soil target analyte concentration is less than the PAL for that analyte (or group of analytes), then the soil contamination has been delineated for that analyte (or group of analytes) and will be evaluated in the SI and/or EE/CA.

11.4.3 Groundwater Decision Rule

- If groundwater target analyte concentration does not exceed their PALs for target analytes, collected below the Fire Fighter Training Pit or the 3 foot step out, then NFA for soils will be recommended.
- If any groundwater target analyte concentration exceeds the PAL for that target analyte, then additional data will be collected via step out samples.
- If any groundwater target analyte concentration is less than the PAL for that analyte (or group of analytes), then the groundwater contamination has been delineated for that analyte (or group of analytes) and will be evaluated in the SI and/or EE/CA.

11.4.4 SI and EE/CA Decision Rule

- If soil and groundwater samples do not exceed their PALs, then recommend a NFA decision.
- If soil and/or groundwater exceed their PALs then, based on the petroleum or CERCLA path decision, conduct additional assessment and/or a Remedial Action Plan (RAP) under Chapter 62-770, F.A.C., or an RI and/or EE/CA under Chapter 62-780, F.A.C.
- The decision to conduct additional assessment and/or a RAP or RI or EE/CA will be evaluated based on the following:

    - Media affected (soil and/or groundwater).
    - Horizontal and vertical extent of contamination in affected media.
    - Magnitude of contamination in affected media.
    - Cost of RI versus EE/CA.

11.5 MEASUREMENT AND PERFORMANCE CRITERIA

Because the biased sampling locations were strategically selected, probability limits for false positive and false negative decision errors were not established. Simple comparisons of measured concentrations to action levels are being used for the first stages of decision making. The Project Team will use the measured results to determine whether the amount and type of data collected are sufficient to support the attainment of the project objectives. This will involve an evaluation of contaminant concentrations and an evaluation of uncertainty for contaminants that have action levels that are less than the laboratory detection limits (LDLs), Limits of Detection (LODs), and LOQs to ensure that contaminants are likely to have been detected, if present. If all data have been collected as planned and no data points are missing or rejected for quality reasons, then the sampling event completeness will be considered satisfactory. If any data gaps are identified, including missing or rejected data, the Project Team will assess whether a claim of having obtained project objectives is reasonable. This assessment will depend on the number and type of identified data gaps; therefore, a more detailed strategy cannot be presented. All Project Team members will be involved in rendering the final conclusion regarding adequacy of the data.

11.6 PLAN FOR OBTAINING DATA

The soil and groundwater sampling design, rationale, and locations are summarized in Worksheets #17 and #18. These worksheets identify the locations that are to be sampled and the analyses to be conducted.

SAP Worksheet #12 -- Measurement Performance Criteria Table – Field Quality Control (QC) Samples

(UFP-QAPP Manual Section 2.6.2)

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria (MPCs)	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Equipment Rinsate Blanks	All Fractions	One per 20 field samples per matrix per sampling equipment[1].	Accuracy/Bias/ Contamination	No analytes ≥ ½ LOQ, except common laboratory contaminants, which must be < LOQ.	S&A
Trip Blanks	VOCs	One per cooler containing VOC samples.	Accuracy/Bias/ Contamination	No analytes ≥ ½ LOQ, except common laboratory contaminants, which must be < LOQ.	S&A
Field Duplicate	All Fractions	One per 10 field samples collected.	Precision	Values > 5X LOQ: Relative Percent Difference (RPD) must be ≤ 30% (aqueous samples) and ≤ 50% (solids)[2, 3].	S&A
Cooler Temperature Indicator	All Fractions	One per cooler.	Representativeness	Temperature must be between 0 and 6 degrees Celsius (°C).	S

1 – Equipment rinsate blanks will be collected if non-dedicated sampling equipment is used.
2 – If duplicate values for non-metals are < 5x LOQ, the absolute difference should be < 2x LOQ.
3 – If duplicate values for metals are < 5x LOQ, the absolute difference should be < 4x LOQ.

SAP Worksheet #13 -- Secondary Data Criteria and Limitations Table
(UFP-QAPP Manual Section 2.7)

Secondary Data	**Data Source** (originating organization, report title and date)	**Data Generator(s)** (originating organization, data types, data generation / collection dates)	**How Data Will Be Used**	**Limitations on Data Use**
None	NA	NA	NA	NA

SAP Worksheet #14 -- Summary of Project Tasks
(UFP-QAPP Manual Section 2.8.1)

The field tasks are summarized below. A short description of these tasks is also provided.

- Mobilization/Demobilization
- Utility Clearance
- Monitoring Equipment Calibration
- MIP/LIF Probe Sampling
- DPT Groundwater Sampling
- Well Installations
- Groundwater Sampling
- Water Level Measurements
- Investigation Derived Waste (IDW) Management
- Field Decontamination Procedures
- Field Documentation Procedures

Additional project activities include the following tasks:

- Analytical Tasks
- Data Management
- Data Review
- Project Reports

Mobilization/Demobilization

Mobilization shall consist of the delivery of all equipment, materials, and supplies to the site; the complete assembly in satisfactory working order of all such equipment at the site; and the satisfactory storage at the site of all such materials and supplies. Tetra Tech will coordinate with the Facility to identify locations for the storage of equipment and supplies. Site-specific Health and Safety Training for all Tetra Tech subcontractors will be provided as part of the site mobilization.

Demobilization shall consist of the prompt and timely removal of all equipment, materials, and supplies from the site following completion of the work. Demobilization includes the cleanup and removal of IDW generated during the conduct of the investigation.

Utility Clearance

Prior to the commencement of any intrusive activities, Tetra Tech will coordinate utility clearance with the Facility and Sunshine State One Call. The Facility and utility companies subscribed to Sunshine State One Call will identify and mark-out utilities that may be present within the proposed well installation areas. Subsurface utilities will also be cleared by the well installation subcontractor by notifying the Sunshine State One Call utility clearing service. See Tetra Tech SOP HS-1.0 (see Appendix A) on conducting well installations for further information.

Monitoring Equipment Calibration

These procedures are described in Worksheet #22.

MIP/LIF Probe Sampling

A MIP/LIF probe will be used to collect field measurements of VOCs in subsurface soil. The readings of VOCs will be accomplished by advancing (typically at a rate of 1 foot per minute) a MIP/LIF detector into the subsurface via DPT methods continuously to target depths. A heating block and the tip of the probe will volatilize VOCs in the subsurface, which will defuse across a semi-permeable membrane to be withdrawn via a carrier gas to the surface where continuous measurements will be made via an ECD and a FID. The fiber optic-based LIF sensor system excites two or three ring aromatic compounds, or PAHs, in the soil adjacent to a sapphire window causing them to fluoresce. The induced fluorescence from the PAHs is returned over a second fiber to the surface where it is quantified using a detector system. The peak wavelength and intensity provide information about the type of petroleum product. Readings will be logged and a computer log generated for each sample location providing VOC/PAH distributions with depth in the subsurface.

DPT Groundwater Sampling

DPT methods will be used to collect in situ groundwater samples at select locations within Site 2. The actual locations and depths will be determined after a review of MIP/LIF data. Groundwater samples will be analyzed in the field by a mobile laboratory (KB Labs) for VOCs. This data will be used to characterize the nature and extent of VOCs that may be present in groundwater due to the fire fighting training activities.

DPT methods involve the advancement of a DPT groundwater sampling screen to a target depth. The screen is then revealed to the formation and groundwater is withdrawn via polyethylene tubing to the

surface via a peristaltic pump. Groundwater samples will be collected via the straw method, placed into vials, and provided to the mobile laboratory (KB Labs) for analysis of VOCs.

Well Installations

Based on the results of MIP/LIF and DPT groundwater sampling, a second phase of field work will be conducted to install monitoring wells. The locations, number, and design of the wells will be determined after review of the MIP/LIF and DPT data. In general, the monitoring wells will be designed to confirm MIP/LIF and field screening results and to provide a sufficient monitoring well network for the evaluation of Site 2. Drilling methods and procedures to install the monitoring wells will follow standard industry practices in accordance with Navy requirements for monitoring well design and installation (NAVFAC SE, 1997). SOPs are detailed in Worksheets #18 and #21.

Groundwater Sampling

Groundwater samples from monitoring wells will be collected using low-flow purging techniques (discharge rate of less than 1 liter per minute) with a peristaltic pump (or submersible pump if the groundwater depth is greater than 20 feet bgs) using Teflon™ tubing dedicated to each well. The monitoring well groundwater samples will be collected using the procedures specified in FS 2200, Groundwater Sampling (FDEP, 2008). Worksheets #17 and #18 specify the groundwater sample locations and analytical groups to be analyzed for each sample for this investigation. Worksheet #23 specifies the analytical methods to be used.

Prior to groundwater sample collection, the monitoring wells will be purged. Both purging and sampling operations will be conducted at a flow rate that results in a groundwater turbidity measurement of 20 Nephelometric Turbidity Units (NTUs) or less (inherent turbidity will be minimized to the greatest extent possible using low flow techniques; individual well conditions and local geology may preclude meeting the 20-NTU criteria) in which case it will be noted in the field logbook and sampling will proceed.

The sample aliquots for SVOCs (including measurements for low level PAHs), pesticides, PCBs, TRPH, and metals will be collected directly from the discharge side of the peristaltic pump following the quiescent sampling procedure. The sample aliquot for VOC analysis will be collected last by slowly pulling the Teflon™ tubing out of the well to minimize agitation of the water in the monitoring well and then transferring the contents of the tubing to a VOC vial. After collection, the samples will be placed in a cooler, chilled with ice, and shipped under chain-of-custody protocol to Katahdin for analysis. SOPs are detailed in Worksheets #18 and #21.

Water Level Measurements

One synoptic round of electronic water-level measurements will be conducted at Site 2 as part of each groundwater sampling event to provide information regarding groundwater flow patterns and hydraulic gradients. Water-level measurements will be completed within the shortest time possible on the same day and no sooner than 24 hours after a significant precipitation event to minimize the precipitation effects on the data sets. Water level measurements will be recorded to the nearest 0.01 foot and referenced to a top of casing notch or northern side of the well casing. The measurement instrument will be decontaminated prior to conducting the measurement event and between each monitoring well. SOPs are detailed in Worksheets #18 and #21.

IDW Management

Types of IDW generated during this investigation that could be potentially contaminated include excess soil material collected but not placed in the laboratory supplied sample jars, sampling equipment decontamination wastewaters, and personnel protective equipment (PPE) and clothing. Based on the historical site activities and types of contaminants present, none of these IDW materials is expected to present a significant risk to human health or the environment if properly managed. Excess soil will initially be placed in 55-gallon labeled, sealable steel drums. The drums will be transported to a secured area designated by the Navy. Proper disposal of these wastes will be performed by the Navy (or its designee) after the analytical results of the soil samples are received from the laboratory and reviewed. PPE and clothing will be wiped clean and disposed of in trash containers.

Field Decontamination Procedure

Decontamination of major equipment and sampling equipment will be in general accordance with FC 1000, Cleaning / Decontamination Procedures (FDEP, 2008).

Field Documentation Procedures

Pre-preserved, certified-clean bottle ware will be supplied by the laboratories. Matrix-specific sample log sheets will be maintained for each sample collected. In addition, sample collection information will be recorded in bound field notebooks or specific field forms. Samples will be packaged and shipped according to FS 1000, General Sampling Procedures (FDEP, 2008).

Field documentation will be performed in accordance with Tetra Tech SOP SA-6.3 (see Appendix A). A summary of all field activities will be properly recorded in indelible ink in a bound logbook with

consecutively numbered pages that cannot be removed. Logbooks will be assigned to field personnel and will be stored in a secured area when not in use.

At a minimum, the following information will be recorded in the site logbook:

- Name of the person to whom the logbook is assigned.
- Project name.
- Project start date.
- Names and responsibilities of on-site project personnel including subcontractor personnel.
- Arrival/departure of site visitors.
- Arrival/departure of equipment.
- Sampling activities and sample log sheet references.
- Description of subcontractor activities.
- Sample pick-up information including chain-of-custody numbers, air bill numbers, carrier, time, and date.
- Description of borehole or monitoring well installation activities and operations.
- Health and safety issues.
- Description of photographs including date, time, photographer, roll and picture number, location, and compass direction of photograph.

All entries will be written in indelible ink and no erasures will be made. If an incorrect entry is made, striking a single line through the incorrect information will make the correction; the person making the correction will initial and date the change.

Analytical Tasks

Chemical analysis will be performed by a mobile laboratory (KB Labs) and a fixed-base laboratory (Katahdin).

KB Labs is a current NELAP-approved laboratory with the State of Florida Department of Health. A copy of the laboratory certification for KB Labs can be found in Appendix B. Analyses will be performed in accordance with the analytical methods identified in Worksheet #19. KB Labs is expected to meet the PALs for VOCs to the extent identified in Worksheet #15. KB Labs will perform chemical analysis following laboratory-specific SOPs (Worksheets #19 and #23) developed based on the analytical methods listed in Worksheets #19 and #30. Replicates consisting of a minimum of 5 percent and a maximum of 5 groundwater samples screened for VOCs by the on-site laboratory will be analyzed off-site by Katahdin for definitive VOCs analysis. At a minimum, the replicates submitted for fixed base laboratory analysis

will represent low medium and high concentrations of the target analytes (e.g., low would be a concentration at or below the detection limit for the target analytes detected by the mobile laboratory, medium would be at the approximate CTLs for the target analytes detected by the mobile laboratory and high would be a concentrations that exceeds the CTLs for the target analytes detected by the mobile laboratory.

Chemical analysis will be performed off-site by Katahdin. Katahdin is accredited under the DoD ELAP. In addition, Katahdin holds NELAP accreditation with the State of Florida Department of Health serving as their primary accrediting authority. Copies of the laboratory accreditation are located in Appendix B. Analyses will be performed in accordance with the analytical methods identified in Worksheet #19. Katahdin will meet the PALs as shown in Worksheet #15. Katahdin will perform chemical analysis following laboratory specific SOPs (Worksheets #19 and #23) developed based on the analytical methods listed in Worksheet #19 and #30. Copies of the laboratory's SOPs are included in Appendix B.

All soil results will be reported by the laboratory on a dry-weight basis. Results of percent moisture will be reported in each analytical data package and electronic data files. This information will also be captured in the project database which will eventually be uploaded to the Naval Installation Restoration Information Solution database. Percent moisture information will also be captured in the SI report.

Data Management

Data Handling and Management – After the field investigation is completed, the field sampling log sheets will be organized by date and media and filed in the project files. The field logbooks for this project will be used only for these sites and will also be categorized and maintained in the project files after the completion of the field program. Project personnel completing concurrent field sampling activities may maintain multiple field logbooks. When possible, logbooks will be segregated by sampling activity. The field logbooks will be titled based on date and activity. The data handling procedures to be followed by the laboratories will meet the requirements of the technical specification. The electronic data results will be automatically downloaded into the Tetra Tech database in accordance with proprietary Tetra Tech processes.

Data Tracking and Control – The Tetra Tech PM (or designee) is responsible for the overall tracking and control of data generated for the project.

- Data Tracking: Data is tracked from its generation to its archiving in the Tetra Tech project-specific files. The Tetra Tech Project Chemist (or designee) is responsible for tracking the samples collected and shipped to the subcontracted laboratory. Upon receipt of the data packages from the analytical

laboratory, the Tetra Tech Project Chemist will oversee the data validation effort, which includes verifying that the data packages are complete and results for all samples have been delivered by the analytical laboratory.

- Data Storage, Archiving, and Retrieval: The data packages received from the subcontracted laboratory are tracked in the data validation logbook. After the data are validated, the data packages are entered into the Tetra Tech CLEAN file system and archived in secure files. The field records including field logbooks, sample logs, chain-of-custody records, and field calibration logs will be submitted by the Tetra Tech FOL to be entered into the CLEAN file system prior to archiving in secure project files. The project files are audited for accuracy and completeness. At the completion of the Navy contract the records will be stored by Tetra Tech and eventually delivered to NAVFAC SE.

- Data Security: The Tetra Tech project files are restricted to designated personnel only. Records can only be borrowed temporarily from the project file using a sign-out system. The Tetra Tech Data Manager maintains the electronic data files. Access to the data files is restricted to qualified personnel only. File and data backup procedures are routinely performed.

Assessment and Oversight – Refer to Worksheet #32 for assessment findings and corrective actions and Worksheet #33 for QA management reports.

Data Review

Data verification is described in Worksheet #34. Data validation is described in Worksheets #35 and #36. Usability assessment is described in Worksheet #37.

Project Reports

An SI report will be prepared summarizing the results of all field activities and presenting all information collected. The SI report will be provided to the Project Team for review. After incorporation of Project Team comments, a final SI report will be prepared and submitted to the Navy, FDEP, and USEPA.

SAP Worksheet #15 -- Reference Limits and Evaluation Table
(UFP-QAPP Manual Section 2.8.1)

Matrix: Soil
Analytical Group: VOCs

Analyte	CAS Number	PAL (mg/kg)	PAL Reference	PQLG (mg/kg)	Katahdin		
					LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
1,1,1-TRICHLOROETHANE	71-55-6	1.9	FDEP Leachability SCTL	0.63	0.005	0.0025	0.00042
1,1,2,2-TETRACHLOROETHANE	**79-34-5**	**0.001**	**FDEP Leachability SCTL**	**0.00033**	**0.005**	**0.0025**	**0.00084**
1,1,2-TRICHLOROETHANE	79-00-5	0.03	FDEP Leachability SCTL	0.010	0.005	0.0025	0.00097
1,1,2-TRICHLOROTRIFLUOROETHANE	76-13-1	11,000	FDEP Leachability SCTL	3.700	0.005	0.0025	0.0009
1,1-DICHLOROETHANE	75-34-3	0.4	FDEP Leachability SCTL	0.13	0.005	0.0025	0.0017
1,1-DICHLOROETHENE	75-35-4	0.06	FDEP Leachability SCTL	0.020	0.005	0.0025	0.00093
1,2-DIBROMO-3-CHLOROPROPANE	**96-12-8**	**0.001**	**FDEP Leachability SCTL**	**0.00033**	**0.005**	**0.0025**	**0.0015**
1,2-DIBROMOETHANE	**106-93-4**	**0.0001**	**FDEP Leachability SCTL**	**0.000033**	**0.005**	**0.0025**	**0.0012**
1,2-DICHLOROBENZENE	95-50-1	17	FDEP Leachability SCTL	5.7	0.005	0.0025	0.00078
1,2-DICHLOROETHANE	107-06-2	0.01	FDEP Leachability SCTL	0.0033	0.005	0.0025	0.001
1,2-DICHLOROPROPANE	78-87-5	0.03	FDEP Leachability SCTL	0.010	0.005	0.0025	0.0014
1,3-DICHLOROBENZENE	541-73-1	7	FDEP Leachability SCTL	2.3	0.005	0.0025	0.00062
1,4-DICHLOROBENZENE	106-46-7	2.2	FDEP Leachability SCTL	0.73	0.005	0.0025	0.00044
2-BUTANONE	78-93-3	17	FDEP Leachability SCTL	5.7	0.025	0.0125	0.0059
2-HEXANONE	591-78-6	1.4	FDEP Leachability SCTL	0.47	0.025	0.0125	0.0048
4-METHYL-2-PENTANONE	108-10-1	2.6	FDEP Leachability SCTL	0.87	0.025	0.0125	0.0059
ACETONE	67-64-1	25	FDEP Leachability SCTL	8.3	0.025	0.0125	0.0051
BENZENE	71-43-2	0.007	FDEP Leachability SCTL	0.0023	0.005	0.0025	0.00092
BROMODICHLOROMETHANE	**75-27-4**	**0.004**	**FDEP Leachability SCTL**	**0.0013**	**0.005**	**0.0025**	**0.0006**
BROMOFORM	75-25-2	0.03	FDEP Leachability SCTL	0.010	0.005	0.0025	0.0007
BROMOMETHANE	74-83-9	0.05	FDEP Leachability SCTL	0.017	0.01	0.005	0.0011
CARBON DISULFIDE	75-15-0	5.6	FDEP Leachability SCTL	1.8	0.005	0.0025	0.00078
CARBON TETRACHLORIDE	56-23-5	0.04	FDEP Leachability SCTL	0.013	0.005	0.0025	0.0013
CHLOROBENZENE	108-90-7	1.3	FDEP Leachability SCTL	0.43	0.005	0.0025	0.00051
CHLORODIBROMOMETHANE	**124-48-1**	**0.003**	**FDEP Leachability SCTL**	**0.0010**	**0.005**	**0.0025**	**0.001**
CHLOROETHANE	75-00-3	0.06	FDEP Leachability SCTL	0.020	0.01	0.005	0.0013
CHLOROFORM	67-66-3	0.29	USEPA R-RSL	0.097	0.005	0.0025	0.00035
CHLOROMETHANE	74-87-3	0.01	FDEP Leachability SCTL	0.0033	0.01	0.005	0.0014
CIS-1,2-DICHLOROETHENE	156-59-2	0.4	FDEP Leachability SCTL	0.13	0.005	0.0025	0.00091
CIS-1,3-DICHLOROPROPENE	10061-01-5	1.7	USEPA R-RSL	0.57	0.005	0.0025	0.00072
CYCLOHEXANE	110-82-7	7,000	USEPA R-RSL	2,300	0.005	0.0025	0.0014

Analyte	CAS Number	PAL (mg/kg)	PAL Reference	PQLG (mg/kg)	Katahdin		
					LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
DICHLORODIFLUOROMETHANE	75-71-8	44	FDEP Leachability SCTL	15	0.01	0.005	0.00092
ETHYLBENZENE	100-41-4	0.6	FDEP Leachability SCTL	0.20	0.005	0.0025	0.00065
ISOPROPYLBENZENE	98-82-8	0.2	FDEP Leachability SCTL	0.067	0.005	0.0025	0.00092
METHYL ACETATE	79-20-9	16	FDEP Leachability SCTL	5.3	0.005	0.0025	0.0027
METHYL TERT-BUTYL ETHER	1634-04-4	0.09	FDEP Leachability SCTL	0.030	0.005	0.0025	0.0011
METHYLENE CHLORIDE	75-09-2	0.02	FDEP Leachability SCTL	0.0067	0.025	0.0125	0.0079
METHYL CYCLOHEXANE	108-87-2	NA	None	NA	0.005	0.0025	0.00096
STYRENE	100-42-5	3.6	FDEP Leachability SCTL	1.2	0.005	0.0025	0.00051
TETRACHLOROETHENE	127-18-4	0.03	FDEP Leachability SCTL	0.010	0.005	0.0025	0.0012
TOLUENE	108-88-3	0.5	FDEP Leachability SCTL	0.17	0.005	0.0025	0.0014
O-XYLENE	95-47-6	3,800	USEPA R-RSL	1,300	0.005	0.0025	0.0013
M+P-XYLENES	TTNUS054	NA	None	NA	0.01	0.005	0.0017
TRANS-1,2-DICHLOROETHENE	95-47-6	0.7	FDEP Leachability SCTL	0.23	0.005	0.0025	0.00071
TRANS-1,3-DICHLOROPROPENE	10061-02-6	1.7	USEPA R-RSL	0.57	0.005	0.0025	0.00086
TRICHLOROETHENE	79-01-6	0.03	FDEP Leachability SCTL	0.010	0.005	0.0025	0.00059
TRICHLOROFLUOROMETHANE	75-69-4	33	FDEP Leachability SCTL	11	0.01	0.005	0.00091
VINYL CHLORIDE	75-01-4	**0.007**	**FDEP Leachability SCTL**	**0.0023**	**0.01**	**0.005**	**0.00087**

Notes:

CAS = Chemical Abstract Service
mg/kg = Milligram per Kilogram
PQLG = Project Quantitation Limit Goal

The PAL references for soil are USEPA Regions 3, 6, and 9 Regional Screening Level, Residential Soil (USEPA, 2010); FDEP Residential SCTL, Chapter 62-777 Residential Soil-Direct, Table II (FDEP, 2005b); FDEP Leachability SCTL: Chapter 62-777 Leachability Based Groundwater, Table II (FDEP, 2005b).

Bolded rows indicate that the PAL is between the laboratory LOQ and the DL. The Project Team has agreed to accept this data for decision making if results below the LOQ are "J" qualified and the results are discussed in the uncertainties section of the Risk Assessment.

Shaded and Bolded rows indicate the PAL is less than the DL; therefore, the Project Team has agreed to replace the PALs with the laboratory LOQs for decision making purposes, as suggested in "Guidance for the Selection of Analytical Methods for the Evaluation of Practical Quantitation Limits" (FDEP, 2004).

Matrix: Soil
Analytical Group: SVOCs and Low Level SVOCs and PAHs

Analyte	CAS Number	PAL (mg/kg)	PAL Reference	PQLG (mg/kg)	Katahdin		
					LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
1-METHYLNAPHTHALENE	90-12-0	3.1	FDEP Leachability SCTL	1.0	0.33	0.25	0.124
1,1-BIPHENYL	**92-52-4**	**0.2**	**FDEP Leachability SCTL**	**0.067**	**0.33**	**0.25**	**0.073**
2,2'-OXYBIS(1-CHLOROPROPANE) (1)	**108-60-1**	**0.009**	**FDEP Leachability SCTL**	**0.0030**	**0.02**	**0.01**	**0.002**
2,4,5-TRICHLOROPHENOL (1)	**95-95-4**	**0.07**	**FDEP Leachability SCTL**	**0.023**	**0.1**	**0.05**	**0.0025**
2,4,6-TRICHLOROPHENOL (1)	**88-06-2**	**0.06**	**FDEP Leachability SCTL**	**0.020**	**0.1**	**0.05**	**0.0033**
2,4-DICHLOROPHENOL (1)	**120-83-2**	**0.003**	**FDEP Leachability SCTL**	**0.0010**	**0.02**	**0.01**	**0.0022**
2,4-DIMETHYLPHENOL	105-67-9	1.7	FDEP Leachability SCTL	0.57	0.33	0.25	0.165
2,4-DINITROPHENOL (1)	**51-28-5**	**0.06**	**FDEP Leachability SCTL**	**0.020**	**0.1**	**0.05**	**0.063**
2,4-DINITROTOLUENE (1)	**121-14-2**	**0.0004**	**FDEP Leachability SCTL**	**0.0013**	**0.02**	**0.01**	**0.007**
2,6-DINITROTOLUENE (1)	**606-20-2**	**0.0004**	**FDEP Leachability SCTL**	**0.0013**	**0.02**	**0.01**	**0.0043**
2-CHLORONAPHTHALENE	91-58-7	260	FDEP Leachability SCTL	87	0.33	0.25	0.083
2-CHLOROPHENOL	95-57-8	0.7	FDEP Leachability SCTL	0.23	0.33	0.25	0.083
2-METHYLNAPHTHALENE	91-57-6	8.5	FDEP Leachability SCTL	2.8	0.33	0.25	0.092
2-METHYLPHENOL	**95-48-7**	**0.3**	**FDEP Leachability SCTL**	**0.10**	**0.33**	**0.25**	**0.2**
2-NITROANILINE	**88-74-4**	**0.1**	**FDEP Leachability SCTL**	**0.033**	**0.82**	**0.62**	**0.075**
2-NITROPHENOL	88-75-5	NA	None	NA	0.33	0.25	0.167
3,3'-DICHLOROBENZIDINE (1)	**91-94-1**	**0.003**	**FDEP Leachability SCTL**	**0.0010**	**0.02**	**0.01**	**0.003**
3-NITROANILINE (1)	**99-09-2**	**0.01**	**FDEP Leachability SCTL**	**0.0033**	**0.02**	**0.01**	**0.0087**
4,6-DINITRO-2-METHYLPHENOL (1)	**534-52-1**	**0.4**	**FDEP Leachability SCTL**	**0.13**	**0.82**	**0.62**	**0.337**
4-BROMOPHENYL PHENYL ETHER	101-55-3	NA	None	NA	0.33	0.25	0.085
4-CHLORO-3-METHYLPHENOL	59-50-7	0.4	FDEP Leachability SCTL	0.13	0.33	0.25	0.166
4-CHLOROANILINE	**106-47-8**	**0.2**	**FDEP Leachability SCTL**	**0.067**	**0.33**	**0.25**	**0.119**
4-CHLOROPHENYL PHENYL ETHER	7005-72-3	NA	None	NA	0.33	0.25	0.078
4-METHYLPHENOL (1)	**106-44-5**	**0.03**	**FDEP Leachability SCTL**	**0.010**	**0.1**	**0.05**	**0.0099**
4-NITROANILINE (1)	**100-01-6**	**0.008**	**FDEP Leachability SCTL**	**0.0027**	**0.02**	**0.01**	**0.0013**
4-NITROPHENOL	**100-02-7**	**0.3**	**FDEP Leachability SCTL**	**0.10**	**0.82**	**0.62**	**0.309**

Analyte	CAS Number	PAL (mg/kg)	PAL Reference	PQLG (mg/kg)	Katahdin		
					LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
ACENAPHTHENE	83-32-9	2.1	FDEP Leachability SCTL	0.70	0.33	0.25	0.065
ACENAPHTHYLENE	208-96-8	27	FDEP Leachability SCTL	9.0	0.33	0.25	0.07
ACETOPHENONE	98-86-2	3.9	FDEP Leachability SCTL	1.3	0.33	0.25	0.178
ANTHRACENE	120-12-7	2,500	Leachability SCTL	830	0.33	0.25	0.084
ATRAZINE	**1912-24-9**	**0.06**	**FDEP Leachability SCTL**	**0.020**	**0.33**	**0.25**	**0.091**
BENZALDEHYDE	100-52-7	4.8	FDEP Leachability SCTL	1.6	0.33	0.25	0.12
BENZO(A)ANTHRACENE	**56-55-3**	**0.15**	**USEPA R-RSL**	**0.050**	0.33	0.25	0.086
BENZO(A)PYRENE (1)	**50-32-8**	**0.015**	**USEPA R-RSL**	**0.0050**	**0.02**	**0.01**	**0.0033**
BENZO(B)FLUORANTHENE	**205-99-2**	**0.15**	**USEPA R-RSL**	**0.050**	0.33	0.25	0.134
BENZO(G,H,I)PERYLENE	191-24-2	1,700	USEPA R-RSL	570	0.33	0.25	0.104
BENZO(K)FLUORA*NTHENE*	207-08-9	1.5	USEPA R-RSL	0.50	0.33	0.25	0.083
BIS(2-CHLOROETHOXY)METHANE	111-91-1	63	FDEP Leachability SCTL	21	0.33	0.25	0.096
BIS(2-CHLOROETHYL)ETHER (1)	**111-44-4**	**0.0001**	**FDEP Leachability SCTL**	**0.000033**	**0.02**	**0.01**	**0.0017**
BIS(2-ETHYLHEXYL)PHTHALATE	117-81-7	35	USEPA R-RSL	12	0.33	0.25	0.098
BUTYL BENZYL PHTHALATE	85-68-7	260	USEPA R-RSL	87	0.33	0.25	0.093
CAPROLACTAM	105-60-2	31,000	USEPA R-RSL	10,000	0.33	0.25	0.144
CARBAZOLE	**86-74-8**	**0.2**	**FDEP Leachability SCTL**	**0.067**	**0.33**	**0.25**	**0.111**
CHRYSENE	218-01-9	15	USEPA R-RSL	5.0	0.33	0.25	0.095
DIBENZO(A,H)ANTHRACENE (1)	**53-70-3**	**0.015**	**SEPA R-RSL**	**0.0050**	**0.02**	**0.01**	**0.0018**
DIBENZOFURAN	132-64-9	15	FDEP Leachability SCTL	5.0	0.33	0.25	0.079
DIETHYL PHTHALATE	84-66-2	86	FDEP Leachability SCTL	29	0.33	0.25	0.08
DIMETHYL PHTHALATE	131-11-3	380	FDEP Leachability SCTL	130	0.33	0.25	0.078
DI-N-BUTYL PHTHALATE	84-74-2	47	FDEP Leachability SCTL	16	0.33	0.25	0.101
DI-N-OCTYL PHTHALATE	117-84-0	1,700	FDEP Residential SCTL	570	0.33	0.25	0.211
FLUORANTHENE	206-44-0	1,200	FDEP Leachability SCTL	400	0.33	0.25	0.106
FLUORENE	86-73-7	160	FDEP Leachability SCTL	53	0.33	0.25	0.081
HEXACHLOROBENZENE	118-74-1	0.3	USEPA R-RSL	0.10	0.33	0.25	0.082
HEXACHLOROBUTADIENE	87-68-3	1	FDEP Leachability SCTL	0.33	0.33	0.25	0.083
HEXACHLOROCYCLOPENTADIENE	77-47-4	9.5	FDEP Residential SCTL	3.2	0.33	0.25	0.082

Analyte	CAS Number	PAL (mg/kg)	PAL Reference	PQLG (mg/kg)	Katahdin		
					LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
HEXACHLOROETHANE	**67-72-1**	**0.2**	**FDEP Leachability SCTL**	**0.067**	**0.33**	**0.25**	**0.096**
INDENO(1,2,3-CD)PYRENE	**193-39-5**	**0.15**	**USEPA R-RSL**	**0.050**	**0.33**	**0.25**	**0.122**
ISOPHORONE	**78-59-1**	**0.2**	**FDEP Leachability SCTL**	**0.067**	**0.33**	**0.25**	**0.075**
NAPHTHALENE	91-20-3	1.2	FDEP Leachability SCTL	0.40	0.33	0.25	0.087
NITROBENZENE	**98-95-3**	**0.02**	**FDEP Residential SCTL**	**0.067**	**0.33**	**0.25**	**0.091**
N-NITROSO-DI-N-PROPYLAMINE (1)	**621-64-7**	**0.00005**	**FDEP Leachability SCTL**	**0.000017**	**0.02**	**0.01**	**0.0019**
N-NITROSODIPHENYLAMINE	86-30-6	0.4	FDEP Leachability SCTL	0.13	0.33	0.25	0.219
PENTACHLOROPHENOL	**87-86-5**	**0.03**	**FDEP Leachability SCTL**	**0.010**	**0.1**	**0.05**	**0.014**
PHENANTHRENE	85-01-8	250	FDEP Leachability SCTL	83	0.33	0.25	0.083
PHENOL (1)	**108-95-2**	**0.05**	**FDEP Leachability SCTL**	**0.017**	**0.1**	**0.05**	**0.0061**
PYRENE	129-00-0	880	FDEP Leachability SCTL	290	0.33	0.25	0.101

Notes:

(1) 8270D Low Level SOP utilized for select SVOCs and PAHs by Selected Ion Monitoring (SIM).

The PAL references for soil are USEPA Regions 3, 6, and 9 Regional Screening Level, Residential Soil (USEPA, 2010); FDEP Residential Soil SCTL (FDEP, 2005b): FL-SCTL 62-777 Residential Soil-Direct, Table II (FDEP, 2005b); FDEP Leachability SCTL: FL-SCTL 62-777 Leachability Based Groundwater, Table II (FDEP, 2005b).

Bolded rows indicate that the PAL is between the laboratory LOQ and the DL. The Project Team has agreed to accept this data for decision making if results below the LOQ are "J" qualified and the results are discussed in the uncertainties section of the Risk Assessment.

Shaded and Bolded rows indicate the PAL is less than the DL; therefore, the Project Team has agreed to replace the PALs with the laboratory LOQs for decision making purposes, as suggested in "Guidance for the Selection of Analytical Methods for the Evaluation of Practical Quantitation Limits" (FDEP, 2004).

Matrix: Soil
Analytical Group: Pesticides

Analyte	CAS Number	PAL (mg/kg)	PAL Reference	PQLG (mg/kg)	Katahdin		
					LOQ (mg/kg)	LOD (mg/kg)	LDL (mg/kg)
4,4'-DDD	72-54-8	2	USEPA R-RSL	0.67	0.0017	0.00085	0.0002
4,4'-DDE	72-55-9	1.4	USEPA R-RSL	0.47	0.0017	0.00085	0.00019
4,4'-DDT	50-29-3	1.7	USEPA R-RSL	0.57	0.0017	0.00085	0.00031
ALDRIN	309-00-2	0.029	USEPA R-RSL	0.0097	0.0017	0.00085	0.00028
ALPHA-BHC	**319-84-6**	**0.0003**	**FDEP Leachability SCTL**	**0.00010**	**0.0017**	**0.00085**	**0.00034**
ALPHA-CHLORDANE	5103-71-9	1.6	USEPA R-RSL	0.53	0.0017	0.00085	0.00021
BETA-BHC	**319-85-7**	**0.001**	**FDEP Leachability SCTL**	**0.00033**	**0.0017**	**0.00085**	**0.00033**
DELTA-BHC	319-86-8	0.077	USEPA R-RSL	0.026	0.0017	0.00085	0.00032
DIELDRIN	60-57-1	0.002	FDEP Leachability SCTL	0.00067	0.0017	0.00085	0.00022
ENDOSULFAN I	959-98-8	370	USEPA R-RSL	120	0.0017	0.00085	0.00024
ENDOSULFAN II	33213-65-9	370	USEPA R-RSL	120	0.0017	0.00085	0.00034
ENDOSULFAN SULFATE	1031-07-8	370	USEPA R-RSL	120	0.0017	0.00085	0.00058
ENDRIN	72-20-8	1	FDEP Leachability SCTL	0.33	0.0017	0.00085	0.00085
ENDRIN ALDEHYDE	7421-93-4	1	FDEP Leachability SCTL	6.0	0.0017	0.00085	0.00049
ENDRIN KETONE	53494-70-5	1	FDEP Leachability SCTL	6.0	0.0017	0.00085	0.00049
GAMMA-BHC (LINDANE)	58-89-9	0.009	FDEP Leachability SCTL	0.0030	0.0017	0.00085	0.00027
GAMMA-CHLORDANE	5103-74-2	1.6	USEPA R-RSL	0.53	0.0017	0.00085	0.00023
HEPTACHLOR	76-44-8	0.11	USEPA R-RSL	0.037	0.0017	0.00085	0.00029
HEPTACHLOR EPOXIDE	1024-57-3	0.053	USEPA R-RSL	0.018	0.0017	0.00085	0.00022
METHOXYCHLOR	72-43-5	160	FDEP Leachability SCTL	53	0.017	0.0085	0.0005
TOXAPHENE	8001-35-2	0.44	USEPA R-RSL	0.15	0.033	0.0165	0.007

Notes:

DDD = Dichlorodiphenyldichloroethane
DDE = Dichlorodiphenyldichloroethylene
DDT = Dichlorodiphenyltrichloroethane
BHC = Benzene hexachloride

The PAL references for soil are USEPA R-RSL: USEPA Regions 3, 6, and 9 Regional Screening Level, Residential Soil (USEPA, 2010); FDEP Residential SCTL: FL-SCTL 62-777 Residential Soil-Direct, Table II (FDEP); FDEP Leachability SCTL (FDEP 2005b): FL-SCTL 62-777 Leachability Based Groundwater, Table II (FDEP, 2005b).

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Matrix: Soil
Analytical Group: PCBs

Analyte	CAS Number	PAL (mg/kg)	PAL Reference	PQLG (mg/kg)	Katahdin		
					LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
AROCLOR-1016	12674-11-2	3.9	USEPA R-RSL	1.3	0.017	0.0085	0.006
AROCLOR-1221	11104-28-2	0.14	USEPA R-RSL	0.017	0.017	0.0085	0.0079
AROCLOR-1232	11141-16-5	0.14	USEPA R-RSL	0.017	0.017	0.0085	0.0093
AROCLOR-1242	53469-21-9	0.22	USEPA R-RSL	0.017	0.017	0.0085	0.0058
AROCLOR-1248	12672-29-6	0.22	USEPA R-RSL	0.017	0.017	0.0085	0.0061
AROCLOR-1254	11097-69-1	0.22	USEPA R-RSL	0.017	0.017	0.0085	0.0047
AROCLOR-1260	11096-82-5	0.22	USEPA R-RSL	0.017	0.017	0.0085	0.006

The PAL references for soil are USEPA R-RSL: USEPA Regions 3, 6, and 9 Regional Screening Level, Residential Soil (USEPA, 2010); FDEP Residential SCTL: FL-SCTL 62-777 Residential Soil-Direct, Table II (FDEP, 2005b); FDEP Leachability SCTL: FL-SCTL 62-777 Leachability Based Groundwater, Table II (FDEP, 2005b).

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Matrix: Soil
Analytical Group: Metals

Analyte	CAS Number	PAL (mg/kg)	PAL Reference	PQLG (mg/kg)	Katahdin		
					LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
ALUMINUM (1)	7429-90-5	77,000	USEPA R-RSL	26,000	30	10	2.9
ANTIMONY (1)	7440-36-0	5.4	FDEP Leachability SCTL	1.8	0.8	0.5	0.099
ARSENIC	**7440-38-2**	**0.39**	**USEPA R-RSL**	**0.13**	**0.8**	**0.5**	**0.09**
BARIUM (1)	7440-39-3	120	FDEP Residential SCTL	40	0.5	0.4	0.061
BERYLLIUM (1)	7440-41-7	63	FDEP Leachability SCTL	21	0.5	0.05	0.0095
CADMIUM	7440-43-9	7.5	FDEP Leachability SCTL	2.5	1	0.3	0.0066
CALCIUM (1)	7440-70-2	NA	None	NA	10	8	2.1
CHROMIUM	7440-47-3	38	FDEP Leachability SCTL	13	1.5	0.4	0.1
COBALT (1)	7440-48-4	23	USEPA R-RSL	7.7	3	0.2	0.027
COPPER (1)	7440-50-8	150	FDEP Residential SCTL	50	2.5	1	0.095
IRON (1)	7439-89-6	53,000	FDEP Residential SCTL	18,000	10	8	1.3
LEAD	7439-92-1	400	FDEP Residential SCTL	130	0.5	0.4	0.14
MAGNESIUM (1)	7439-95-4	NA	None	NA	10	8	0.92
MANGANESE (1)	7439-96-5	3,500	FDEP Residential SCTL	600	0.5	0.4	0.14
MERCURY (1)	7439-97-6	2.1	FDEP Leachability SCTL	0.70	0.04	0.005	0.0022
NICKEL (1)	7440-02-0	130	FDEP Leachability SCTL	43	4	0.4	0.033
POTASSIUM (1)	9/7/7440	NA	None	NA	100	40	5.3
SELENIUM (1)	7782-49-2	5.2	FDEP Leachability SCTL	1.7	1	1	0.22
SILVER (1)	7440-22-4	17	FDEP Leachability SCTL	5.7	1.5	0.5	0.065
SODIUM (1)	7440-23-5	NA	None	NA	100	50	3.9
THALLIUM (1)	7440-28-0	2.8	FDEP Leachability SCTL	0.93	1.5	0.5	0.092
VANADIUM (1)	7440-62-2	67	FDEP Residential SCTL	22	2.5	0.4	0.039
ZINC (1)	7440-66-6	23,000	USEPA R-RSL	7,700	2.5	1.2	0.012

The PAL references for soil are USEPA R-RSL: USEPA Regions 3, 6, and 9 Regional Screening Level, Residential Soil (USEPA, 2010); FDEP Residential SCTL: FL-SCTL 62-777 Residential Soil-Direct, Table II (FDEP, 2005b); FDEP Leachability SCTL: FL-SCTL 62-777 Leachability Based Groundwater, Table II (FDEP, 2005b).

(1) Analysis of soil samples is to only consist of the Florida Used Oil Group, arsenic, cadmium, chromium and lead, unless the project proceeds on a path similar to CERCLA or Chapter 62-780, F.A.C. and it is determined that other metals could have been released to the environment based on the assessment findings.

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Matrix: Soil
Analytical Group: TRPH

Analyte	CAS Number	PAL (mg/kg)	PAL Reference	PQLG (mg/kg)	Katahdin		
					LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
TRPH	NA	340	FDEP Leachability SCTL	110	5	2.5	2.6

The PAL references for soil are USEPA R-RSL: USEPA Regions 3, 6, and 9 Regional Screening Level, Residential Soil (USEPA, 2010); FDEP Residential SCTL: FL-SCTL 62-777 Residential Soil-Direct, Table II (FDEP, 2005b); FDEP Leachability SCTL: FL-SCTL 62-777 Leachability Based Groundwater, Table II (FDEP, 2005b).

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Shaded and Bolded rows indicate the PAL is less than the DL; therefore, the Project Team has agreed to replace the PALs with the laboratory LOQs for decision making purposes, as suggested in "Guidance for the Selection of Analytical Methods for the Evaluation of Practical Quantitation Limits" (FDEP, 2004).

Matrix: Groundwater
Analytical Group: VOCs

Analyte	CAS Number	PAL (µg/L)	PAL Reference	PQLG (µg/L)	Katahdin		
					LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
1,1,1-TRICHLOROETHANE	71-55-6	200	FDEP GCTL	67	1	0.5	0.20
1,1,2,2-TETRACHLOROETHANE	**79-34-5**	**0.067**	**USEPA T-RSL**	**0.022**	**1**	**0.5**	**0.38**
1,1,2-TRICHLOROETHANE	**79-00-5**	**0.24**	**USEPA T-RSL**	**0.080**	**1**	**0.5**	**0.33**
1,1,2-TRICHLOROTRIFLUOROETHANE	76-13-1	59,000	USEPA T-RSL	20,000	1	0.5	0.31
1,1-DICHLOROETHANE	75-34-3	2.4	USEPA T-RSL	0.80	1	0.5	0.21
1,1-DICHLOROETHENE	75-35-4	7	FDEP GCTL	2.3	1	0.5	0.35
1,2-DIBROMO-3-CHLOROPROPANE	**96-12-8**	**0.00032**	**USEPA T-RSL**	**0.00011**	**1**	**0.5**	**0.5**
1,2-DIBROMOETHANE	**106-93-4**	**0.0065**	**USEPA T-RSL**	**0.0022**	**1**	**0.5**	**0.22**
1,2-DICHLOROBENZENE	95-50-1	370	USEPA T-RSL	120	1	0.5	0.15
1,2-DICHLOROETHANE	**107-06-2**	**0.15**	**USEPA T-RSL**	**0.050**	**1**	**0.5**	**0.2**
1,2-DICHLOROPROPANE	**78-87-5**	**0.39**	**USEPA T-RSL**	**0.13**	**1**	**0.5**	**0.25**
1,3-DICHLOROBENZENE	541-73-1	210	FDEP GCTL	70	1	0.5	0.26
1,4-DICHLOROBENZENE	**106-46-7**	**0.43**	**USEPA T-RSL**	**0.14**	**1**	**0.5**	**0.24**
2-BUTANONE	78-93-3	4,200	FDEP GCTL	1,400	5	2.5	1.31
2-HEXANONE	591-78-6	47	USEPA T-RSL	16	5	2.5	1.7
4-METHYL-2-PENTANONE	108-10-1	560	FDEP GCTL	190	5	2.5	1.32
ACETONE	67-64-1	6,300	FDEP GCTL	2,100	5	2.5	2.21
BENZENE	**71-43-2**	**0.41**	**USEPA T-RSL**	**0.14**	**1**	**0.5**	**0.26**
BROMODICHLOROMETHANE	**75-27-4**	**0.12**	**USEPA T-RSL**	**0.040**	**1**	**0.5**	**0.33**
BROMOFORM	75-25-2	4.4	FDEP GCTL	1.5	1	0.5	0.23
BROMOMETHANE	74-83-9	8.7	USEPA T-RSL	2.9	2	1	0.49
CARBON DISULFIDE	75-15-0	700	FDEP GCTL	230	1	0.5	0.25
CARBON TETRACHLORIDE	**56-23-5**	**0.2**	**USEPA T-RSL**	**1.0**	**1**	**0.5**	**0.22**
CHLOROBENZENE	108-90-7	91	USEPA T-RSL	30	1	0.5	0.22
CHLORODIBROMOMETHANE	**124-48-1**	**0.15**	**USEPA T-RSL**	**0.050**	**1**	**0.5**	**0.3**
CHLOROETHANE	75-00-3	12	FDEP GCTL	4.0	2	1	0.55
CHLOROFORM	**67-66-3**	**0.19**	**USEPA T-RSL**	**0.063**	**1**	**0.5**	**0.32**
CHLOROMETHANE	74-87-3	2.7	FDEP GCTL	0.90	1	0.5	0.36
CIS-1,2-DICHLOROETHENE	156-59-2	70	FDEP GCTL	23	1	0.5	0.21
CIS-1,3-DICHLOROPROPENE	**10061-01-5**	**0.43**	**USEPA T-RSL**	**0.14**	**1**	**0.5**	**0.19**
CYCLOHEXANE	110-82-7	13,000	USEPA T-RSL	4,300	1	0.5	0.31
DICHLORODIFLUOROMETHANE	75-71-8	390	USEPA T-RSL	130	2	1	0.24
ETHYLBENZENE	100-41-4	1.5	USEPA T-RSL	0.50	1	0.5	0.21

Analyte	CAS Number	PAL (µg/L)	PAL Reference	PQLG (µg/L)	Katahdin		
					LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
ISOPROPYLBENZENE	**98-82-8**	**0.8**	**FDEP GCTL**	**0.27**	**1**	**0.5**	**0.23**
METHYL ACETATE	79-20-9	3,000	FDEP CTL	1,000	1	0.5	0.53
METHYL TERT-BUTYL ETHER	1634-04-4	12	USEPA T-RSL	4.0	1	0.5	0.36
METHYLENE CHLORIDE	**75-09-2**	**4.8**	**USEPA T-RSL**	**1.6**	**5**	**2.5**	**1.13**
METHYL CYCLOHEXANE	108-87-2	NA	None	NA	1	0.5	0.3
STYRENE	100-42-5	100	FDEP GCTL	33	1	0.5	0.23
TETRACHLOROETHENE	**127-18-4**	**0.11**	**USEPA T-RSL**	**0.037**	**1**	**0.5**	**0.4**
TOLUENE	108-88-3	40	FDEP GCTL	13	1	0.5	0.27
O-XYLENE	95-47-6	1,200	USEPA T-RSL	400	1	0.5	0.25
M+P-XYLENES	TTNUS054	10,000	FDEP Primary Standard	3,300	2	1	0.59
TRANS-1,2-DICHLOROETHENE	156-60-5	100	FDEP RGCTL	33	1	0.5	0.25
TRANS-1,3-DICHLOROPROPENE	10061-02-6	NA	None	NA	1	0.5	0.2
TRICHLOROETHENE	79-01-6	2	USEPA T-RSL	0.67	1	0.5	0.28
TRICHLOROFLUOROMETHANE	75-69-4	1,300	USEPA T-RSL	430	2	1	0.24
VINYL CHLORIDE	75-01-4	0.016	USEPA T-RSL	0.0053	2	1	0.25

Notes:

Note that groundwater samples analyzed by KB Labs in the field will be analyzed for the sample VOCs listed above. Typical KB Lab LOQs are 1.0 µg/L, and LDLs range from 0.5 to 0.2 µg/L.

The PAL references for groundwater are USEPA T-RSL: USEPA Regions 3, 6, and 9 Regional Screening Level, Tap water (USEPA, 2010); FDEP Residential GCTL: FL-GCTL 62-777 Groundwater, Table I (FDEP, 2005b); FDEP Primary Standard: FL-Drinking Water Primary Standard, Chapter 62-550 (FDEP, 2007); FDEP Secondary Standard: FL-Drinking Water Secondary Standard, Chapter 62-550 (FDEP, 2007).

Bolded rows indicate that the PAL is between the laboratory LOQ and the DL. The Project Team has agreed to accept this data for decision making if results below the LOQ are "J" qualified and the results are discussed in the uncertainties section of the Risk Assessment.

Shaded and Bolded rows indicate the PAL is less than the DL; therefore, the Project Team has agreed to replace the PALs with the laboratory LOQs for decision making purposes, as suggested in "Guidance for the Selection of Analytical Methods for the Evaluation of Practical Quantitation Limits" (FDEP, 2004).

Matrix: Groundwater
Analytical Group: SVOCs and Low Level SVOCs and PAHs

Analyte	CAS Number	PAL (µg/L)	PAL Reference	PQLG (µg/L)	Katahdin		
					LOQ (µg/L)	LOD (µg/L)	L (µg/L)
1-METHYLNAPHTHALENE (1)	90-12-0	2.3	USEPA T-RSL	0.77	0.2	0.1	0.068
1,1-BIPHENYL	**92-52-4**	**0.5**	**FDEP GCTL**	**0.17**	**10**	**7.5**	**2.7**
2,2'-OXYBIS(1-CHLOROPROPANE)	**108-60-1**	**0.5**	**FDEP GCTL**	**0.17**	**10**	**7.5**	**2.1**
2,4,5-TRICHLOROPHENOL	**95-95-4**	**1**	**FDEP GCTL**	**0.33**	**25**	**18.75**	**3.6**
2,4,6-TRICHLOROPHENOL	**88-06-2**	**3.2**	**FDEP GCTL**	**1.1**	**10**	**7.5**	**2.7**
2,4-DICHLOROPHENOL	**120-83-2**	**0.3**	**FDEP GCTL**	**0.10**	**10**	**7.5**	**3**
2,4-DIMETHYLPHENOL	105-67-9	140	FDEP GCTL	47	10	7.5	4.4
2,4-DINITROPHENOL	51-28-5	14	FDEP GCTL	4.7	25	18.75	1
2,4-DINITROTOLUENE	**121-14-2**	**0.05**	**FDEP GCTL**	**0.017**	**10**	**7.5**	**2.2**
2,6-DINITROTOLUENE	**606-20-2**	**0.05**	**FDEP GCTL**	**0.017**	**10**	**7.5**	**2**
2-CHLORONAPHTHALENE	91-58-7	560	FDEP GCTL	190	10	7.5	2.9
2-CHLOROPHENOL	95-57-8	35	FDEP GCTL	12	10	7.5	3.2
METHYLNAPHTHALENE (1)	91-57-6	28	FDEP GCTL	9.3	0.2	0.1	0.077
2-METHYLPHENOL	95-48-7	35	FDEP GCTL	12	10	7.5	3.8
2-NITROANILINE	**88-74-4**	**21**	**FDEP GCTL**	**7.0**	**25**	**18.75**	**1.8**
2-NITROPHENOL (2)	88-75-5	56	FDEP GCTL	19	10	7.5	2.7
3,3'-DICHLOROBENZIDINE	**91-94-1**	**0.08**	**FDEP GCTL**	**0.028**	**10**	**7.5**	**1.1**
3-NITROANILINE	**99-09-2**	**1.7**	**FDEP GCTL**	**0.57**	**25**	**18.75**	**1.5**
4,6-DINITRO-2-METHYLPHENOL	**534-52-1**	**3.7**	**USEPA T-RSL**	**1.2**	**25**	**18.75**	**2**
4-BROMOPHENYL PHENYL ETHER	101-55-3	NA	None	NA	10	7.5	1.9
4-CHLORO-3-METHYLPHENOL	59-50-7	63	FDEP GCTL	21	10	7.5	3.6
4-CHLOROANILINE	106-47-8	0.34	**USEPA T-RSL**	0.11	10	7.5	1.9
4-CHLOROPHENYL PHENYL ETHER	7005-72-3	NA	None	NA	10	7.5	2.2
4-METHYLPHENOL	**106-44-5**	**3.5**	**FDEP GCTL**	**1.2**	**10**	**7.5**	**5.6**
4-NITROANILINE	**100-01-6**	**1.7**	**FDEP GCTL**	**0.57**	**25**	**18.75**	**1.6**
4-NITROPHENOL	100-02-7	56	FDEP GCTL	19	25	18.75	1.8
ACENAPHTHENE (1)	83-32-9	20	FDEP GCTL	6.7	0.2	0.1	0.064

Analyte	CAS Number	PAL (µg/L)	PAL Reference	PQLG (µg/L)	Katahdin		
					LOQ (µg/L)	LOD (µg/L)	L (µg/L)
ACENAPHTHYLENE (1)	208-96-8	210	FDEP GCTL	70	0.2	0.1	0.054
ACETOPHENONE	98-86-2	700	FDEP GCTL	230	10	7.5	3.9
ANTHRACENE (1)	120-12-7	2,100	FDEP GCTL	700	0.2	0.1	0.044
ATRAZINE	**1912-24-9**	**0.29**	**USEPA T-RSL**	**0.097**	**10**	**7.5**	**3.3**
BENZALDEHYDE	100-52-7	700	FDEP GCTL	230	10	7.5	1
BENZO(A)ANTHRACENE (1	**56-55-3**	**0.029**	**USEPA T-RSL**	**0.0097**	**0.2**	**0.1**	**0.046**
BENZO(A)PYRENE (1)	**50-32-8**	**0.0029**	**USEPA T-RSL**	**0.00097**	**0.2**	**0.1**	**0.066**
BENZO(B)FLUORANTHENE (1)	**205-99-2**	**0.029**	**USEPA T-RSL**	**0.0097**	**0.2**	**0.1**	**0.089**
BENZO(G,H,I)PERYLENE (1)	191-24-2	210	FDEP GCTL	70	0.2	0.1	0.065
BENZO(K)FLUORANTHENE (1)	207-08-9	0.29	USEPA T-RSL	0.097	0.2	0.1	0.049
BIS(2-CHLOROETHOXY)METHANE	111-91-1	110	USEPA T-RSL	37	10	7.5	2.1
BIS(2-CHLOROETHYL)ETHER	**111-44-4**	**0.012**	**USEPA T-RSL**	**0.0040**	**10**	**7.5**	**2**
BIS(2-ETHYLHEXYL)PHTHALATE	**117-81-7**	**4.8**	**USEPA T-RSL**	**1.6**	**10**	**0.5**	**1.8**
BUTYL BENZYL PHTHALATE	85-68-7	35	USEPA T-RSL	12	10	7.5	1.9
CAPROLACTAM	105-60-2	18,000	USEPA T-RSL	6,000	10	7.5	0.4
CARBAZOLE	**86-74-8**	**1.8**	**FDEP GCTL**	**0.60**	**10**	**7.5**	**2.1**
CHRYSENE (1)	218-01-9	2.9	USEPA T-RSL	0.97	0.2	0.1	0.036
DIBENZO(A,H)ANTHRACENE (1)	**53-70-3**	**0.0029**	**USEPA T-RSL**	**0.00097**	**0.2**	**0.1**	**0.07**
DIBENZOFURAN	132-64-9	28	FDEP GCTL	9.3	10	7.5	1.6
DIETHYL PHTHALATE	84-66-2	5,600	FDEP GCTL	1,900	10	7.5	2
DIMETHYL PHTHALATE	131-11-3	70,000	FDEP GCTL	23,000	10	7.5	2
DI-N-BUTYL PHTHALATE	84-74-2	700	FDEP GCTL	230	10	7.5	2.5
DI-N-OCTYL PHTHALATE	117-84-0	140	FDEP GCTL	47	10	7.5	1.8
FLUORANTHENE (1)	206-44-0	280	FDEP GCTL	93	0.2	0.1	0.073
FLUORENE (1)	86-73-7	280	FDEP GCTL	93	0.2	0.1	0.061
HEXACHLOROBENZENE	**118-74-1**	**0.042**	**USEPA T-RSL**	**0.014**	**10**	**7.5**	**2.1**
HEXACHLOROBUTADIENE	**87-68-3**	**0.4**	**FDEP Residential GCTL**	**0.13**	**10**	**7.5**	**1.8**
HEXACHLOROCYCLOPENTADIENE	77-47-4	50	FDEP GCTL	17	10	7.5	1.2
HEXACHLOROETHANE	**67-72-1**	**2.5**	**FDEP GCTL**	**0.83**	**10**	**7.5**	**2.3**

Analyte	CAS Number	PAL (µg/L)	PAL Reference	PQLG (µg/L)	Katahdin		
					LOQ (µg/L)	LOD (µg/L)	L (µg/L)
DENO(1,2,3-CD)PYRENE (1)	**193-39-5**	**0.029**	**USEPA T-RSL**	**0.0097**	**0.2**	**0.1**	**0.052**
ISOPHORONE	78-59-1	37	FDEP GCTL	12	10	7.5	1.7
NAPHTHALENE (1)	**91-20-3**	**0.14**	**USEPA T-RSL**	**0.047**	**0.2**	**0.1**	**0.05**
NITROBENZENE	**98-95-3**	**0.12**	**USEPA T-RSL**	**0.040**	**10**	**7.5**	**3.1**
N-NITROSO-DI-N-PROPYLAMINE	**621-64-7**	**0.005**	**FDEP GCTL**	**0.0017**	**10**	**7.5**	**1.9**
N-NITROSODIPHENYLAMINE	86-30-6	7.1	FDEP GCTL	2.7	10	7.5	3.7
PENTACHLOROPHENOL	**87-86-5**	**0.56**	**USEPA T-RSL**	**0.33**	**25**	**18.75**	**2.3**
PHENANTHRENE	85-01-8	210	FDEP GCTL	70	0.2	0.1	0.051
PHENOL	108-95-2	10	FDEP GCTL	3.3	10	7.5	1.8
PYRENE	129-00-0	210	FDEP GCTL	70	0.2	0.1	0.059

Notes:

1. 8270D Low Level SOP utilized for select SVOCs and PAHs by SIM.
2. FDEP Residential GCTL FOR 4-NITROPHENOL

The PAL references for groundwater are USEPA T-RSL: USEPA Regions 3, 6, and 9 Regional Screening Level, Tap Water (USEPA, 2010); FDEP Residential GCTL: FL-GCTL 62-777 Groundwater, Table I (FDEP, 2005b); FDEP Primary Standard: FL-Drinking Water Primary Standard, 62-550 (FDEP, 2007); FDEP Secondary Standard: FL-Drinking Water Secondary Standard, 62-550 (FDEP, 2007).

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Matrix: Groundwater
Analytical Group: Pesticides

Analyte	CAS Number	PAL (µg/L)	PAL Reference	PQLG (µg/L)	Katahdin		
					LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
4,4'-DDD	72-54-8	0.1	FDEP GCTL	0.033	0.01	0.005	0.0018
4,4'-DDE	72-55-9	0.1	FDEP GCTL	0.033	0.01	0.005	0.00098
4,4'-DDT	50-29-3	0.1	FDEP GCTL	0.033	0.01	0.005	0.00178
ALDRIN	**309-00-2**	**0.002**	**FDEP GCTL**	**0.00067**	**0.01**	**0.005**	**0.00148**
ALPHA-BHC	**319-84-6**	**0.006**	**FDEP GCTL**	**0.0020**	**0.01**	**0.005**	**0.00138**
ALPHA-CHLORDANE	5103-71-9	0.19	USEPA T-RSL	0.063	0.01	0.005	0.00152
BETA-BHC	**319-85-7**	**0.02**	**FDEP GCTL**	**0.0067**	**0.01**	**0.005**	**0.00126**
DELTA-BHC	319-86-8	0.011	USEPA T-RSL	0.0037	0.01	0.005	0.0026
DIELDRIN	**60-57-1**	**0.002**	**FDEP GCTL**	**0.00067**	**0.01**	**0.005**	**0.0013**
ENDOSULFAN I	959-98-8	220	USEPA T-RSL	73	0.01	0.005	0.00128
ENDOSULFAN II	33213-65-9	220	USEPA T-RSL	73	0.01	0.005	0.00114
ENDOSULFAN SULFATE	1031-07-8	220	USEPA T-RSL	73	0.01	0.005	0.00134
ENDRIN	72-20-8	2	FDEP GCTL	0.67	0.01	0.005	0.00168
ENDRIN ALDEHYDE	7421-93-4	11	USEPA T-RSL	3.7	0.01	0.005	0.00124
ENDRIN KETONE	53494-70-5	11	USEPA T-RSL	3.7	0.01	0.005	0.00124
GAMMA-BHC (LINDANE)	58-89-9	0.061	USEPA T-RSL	0.020	0.01	0.005	0.00144
GAMMA-CHLORDANE	5103-74-2	0.19	USEPA T-RSL	0.063	0.01	0.005	0.0012
HEPTACHLOR	76-44-8	0.015	USEPA T-RSL	0.050	0.01	0.005	0.0016
HEPTACHLOR EPOXIDE	**1024-57-3**	**0.0074**	**USEPA T-RSL**	**0.0025**	**0.01**	**0.005**	**0.0048**
METHOXYCHLOR	72-43-5	40	FDEP GCTL	13	0.01	0.005	0.00168
TOXAPHENE	8001-35-2	0.061	USEPA T-RSL	0.020	0.02	0.1	0.034

Notes:

The PAL references for groundwater are USEPA T-RSL: USEPA Regions 3, 6, and 9 Regional Screening Level, Tap water (USEPA, 2010); FDEP Residential GCTL: FL-GCTL 62-777 Groundwater, Table I (FDEP, 2005b); FDEP Primary Standard: FL-Drinking Water Primary Standard, 62-550 (FDEP, 2007); FDEP Secondary Standard: FL-Drinking Water Secondary Standard, 62-550 (FDEP, 2007).

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Shaded and Bolded rows indicate the PAL is less than the DL; therefore, the Project Team has agreed to replace the PALs with the laboratory LOQs for decision making purposes, as suggested in "Guidance for the Selection of Analytical Methods for the Evaluation of Practical Quantitation Limits" (FDEP, 2004).

Matrix: Groundwater
Analytical Group: PCBs

Analyte	CAS Number	PAL (µg/L)	PAL Reference	PQLG (µg/L)	Katahdin		
					LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
AROCLOR-1016	12674-11-2	0.5	FDEP Primary Standard	0.17	0.1	0.05	0.03
AROCLOR-1221	**11104-28-2**	**0.0068**	**USEPA T-RSL**	**0.0023**	**0.1**	**0.05**	**0.04**
AROCLOR-1232	**11141-16-5**	**0.0068**	**USEPA T-RSL**	**0.0023**	**0.1**	**0.05**	**0.0178**
AROCLOR-1242	**53469-21-9**	**0.034**	**USEPA T-RSL**	**0.011**	**0.1**	**0.05**	**0.036**
AROCLOR-1248	**12672-29-6**	**0.034**	**USEPA T-RSL**	**0.011**	**0.1**	**0.05**	**0.04**
AROCLOR-1254	**11097-69-1**	**0.034**	**USEPA T-RSL**	**0.011**	**0.1**	**0.05**	**0.0164**
AROCLOR-1260	**11096-82-5**	**0.034**	**USEPA T-RSL**	**0.011**	**0.1**	**0.05**	**0.034**

Notes:

The PAL references for groundwater are USEPA T-RSL: USEPA Regions 3, 6, and 9 Regional Screening Level, Tap water (USEPA, 2010); FDEP Residential GCTL: FL-GCTL 62-777 Groundwater, Table I (FDEP, 2005b); FDEP Primary Standard: FL-Drinking Water Primary Standard, 62-550 (FDEP, 2007); FDEP Secondary Standard: FL-Drinking Water Secondary Standard, 62-550 (FDEP, 2007).

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Shaded and Bolded rows indicate the PAL is less than the DL; therefore, the Project Team has agreed to replace the PALs with the laboratory LOQs for decision making purposes as suggested in "Guidance for the Selection of Analytical Methods for the Evaluation of Practical Quantitation Limits" (FDEP, 2004).

Matrix: Groundwater
Analytical Group: Metals

Analyte	CAS Number	PAL (µg/L)	PAL Reference	PQLG (µg/L)	Katahdin		
					LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
ALUMINUM (2)	7429-90-5	200	FDEP Secondary Standard	17	300	100	13
ANTIMONY (1) (2)	7440-36-0	6	FDEP GCTL	2.0	8	5	1.3
ARSENIC (1)	**7440-38-2**	**0.045**	**USEPA T-RSL**	**0.015**	**8**	**5**	**0.2**
BARIUM (2)	7440-39-3	2,000	FDEP GCTL	670	5	4	0.35
BERYLLIUM (2)	**7440-41-7**	**4**	**FDEP GCTL**	**1.3**	**5**	**0.5**	**0.091**
CADMIUM	**7440-43-9**	**5**	**FDEP GCTL**	**1.7**	**10**	**3**	**0.12**
CALCIUM (2)	7440-70-2	NA	None	NA	100	80	6.8
CHROMIUM	7440-47-3	100	FDEP GCTL	33	15	4	0.44
COBALT (2)	**7440-48-4**	**11**	**USEPA T-RSL**	**3.7**	**30**	**2**	**0.27**
COPPER (2)	7440-50-8	1,000	FDEP GCTL	330	25	10	0.7
IRON (2)	7439-89-6	300	FDEP GCTL	100	100	80	5.2
LEAD (2)	7439-92-1	15	FDEP GCTL	5.0	5	4	1
MAGNESIUM (2)	7439-95-4	NA	None	NA	100	80	9.4
MANGANESE (2)	7439-96-5	50	FDEP GCTL	17	5	4	0.67
MERCURY (2)	7439-97-6	0.57	USEPA T-RSL	0.19	0.2	0.1	0.021
NICKEL (2)	7440-02-0	100	FDEP GCTL	33	40	4	0.24
POTASSIUM (2)	9/7/7440	NA	None	NA	1,000	400	95
SELENIUM (2)	7782-49-2	35	FDEP GCTL	17	10	7	3
SILVER (2)	7440-22-4	35	FDEP GCTL	33	15	5	0.79
SODIUM (2)	7440-23-5	160,000	FDEP GCTL	53,000	1,000	500	22
THALLIUM (1) (2)	**7440-28-0**	**2**	**FDEP Primary Standard**	**0.67**	**15**	**5**	**0.6**
VANADIUM (2)	7440-62-2	49	FDEP GCTL	16	25	4	0.35
ZINC (2)	7440-66-6	2,100	FDEP GCTL	1,700	25	12	1.7

Notes: (1) Low Level Modification (final extract volume = 2 milliliters [mL]) to obtain lower detection limits for these analytes.

(2) Analysis of groundwater samples is to only consist of the Florida Used Oil Group, arsenic, cadmium, chromium and lead, unless the project proceeds on a path similar to CERCLA or Chapter 62-780, F.A.C. and it is determined that other metals could have been released to the environment based on the assessment findings.

The PAL references for groundwater are USEPA T-RSL: USEPA Regions 3, 6, and 9 Regional Screening Level, Tap water (USEPA, 2010); FDEP Residential GCTL: FL-GCTL 62-777 Groundwater, Table I (FDEP, 2005b); FDEP Primary Standard: FL-Drinking Water Primary Standard, 62-550 (FDEP, 2007); FDEP Secondary Standard: FL-Drinking Water Secondary Standard, 62-550 (FDEP, 2007).

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Matrix: Groundwater
Analytical Group: TRPH

Analyte	CAS Number	PAL (mg/kg)	PAL Reference	PQLG (mg/kg)	Katahdin		
					LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
TRPH	NA	5,000	FDEP GCTL	1,700	75	37.5	9.1

Notes:

The PAL references for groundwater are USEPA T-RSL: USEPA Regions 3, 6, and 9 Regional Screening Level, Tap water (USEPA, 2010); FDEP Residential GCTL: FL-GCTL 62-777 Groundwater, Table I (FDEP, 2005b); FDEP Primary Standard: FL-Drinking Water Primary Standard, 62-550 (FDEP, 2007); FDEP Secondary Standard: FL-Drinking Water Secondary Standard, 62-550 (FDEP, 2007).

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SAP Worksheet #16 -- Project Schedule / Timeline Table
(UFP-QAPP Manual Section 2.8.2)

Activities	Organization	Dates		Deliverable
		Anticipated Date(s) of Initiation	Anticipated Date of Completion	
Prepare Draft UFP SAP	Tetra Tech	4/21/10	6/21/10	
Submit Draft UFP SAP	Tetra Tech	6/21/10	6/21/10	Draft UFP SAP
Navy Review	Navy	6/21/10	7/7/10	
Prepare Final Draft UFP SAP	Tetra Tech	7/13/10	7/19/10	
Submit Final Draft UFP SAP	Tetra Tech	7/19/10	7/19/10	Final Draft UFP SAP
Regulator Review	FDEP	7/19/10	9/1/10	
Receive Comments/Comment Resolution	Tetra Tech	9/1/10	9/7/10	
Prepare Final UFP SAP	Tetra Tech	9/8/10	9/16/10	
Submit Final UFP SAP	Tetra Tech	9/16/10	9/16/10	Final UFP SAP
Mobilization and Field Investigation	Tetra Tech	9/20/10	10/1/10	
Complete Field Investigation and Demobilization	Tetra Tech	12/17/10	12/31/10	
Laboratory Analysis	Katahdin and KB Labs	10/1/10	12/31/11	
Data Validation	Tetra Tech	10/4/10	2/28/11	
Database Entry	Tetra Tech	10/4/10	2/28/11	
Prepare Draft SI Report	Tetra Tech	2/28/11	4/29/11	
Submit Draft SI Report	Tetra Tech	4/29/11	4/29/11	Draft SI Report
Navy Review	Navy	4/29/11	5/27/11	
Prepare Final Draft SI Report	Tetra Tech	5/27/11	6/10/11	
Submit Final Draft SI Report	Tetra Tech	6/10/11	6/10/11	Final Draft SI Report
Regulator Review	FDEP	6/10/11	7/8/11	
Receive Comments/Comment Resolution	Tetra Tech	7/8/11	7/22/11	
Prepare Final SI Report	Tetra Tech	7/22/11	7/29/11	
Submit Final SI Report	Tetra Tech	7/29/11	7/29/11	Final SI Report

SAP Worksheet #17 -- Sampling Design and Rationale
(UFP-QAPP Manual Section 3.1.1)

A Triad Approach is being utilized to evaluate the nature and extent of contamination at Site 2. The Triad Approach is an approach to accelerate the decision-making process for hazardous waste site evaluation and cleanup. The approach provides a framework for efficiently using real-time environmental sensors and tools to improve decision-making by systematic project planning (SPP), dynamic work strategies, and innovative rapid sampling and analytical technologies.

This UFP-SAP provides the elements of the SPP stage including the involvement of site stakeholders in the decision making process and the development of DQOs and a CSM. Dynamic work strategies are strategies that incorporate adaptable project activities to site conditions as new information becomes available while work is underway. This allows for optimization of the data collection effort to better eliminate uncertainties and to integrate the site data into the evaluation of potential site remedies. Rapid sampling techniques will be utilized to provide real-time delineation of VOCs in the subsurface at the site via use of the MIP/LIF system combined with DPT groundwater sampling and on-site mobile laboratory analysis. The MIP/LIF provides a real-time three-dimensional visual display of the data as the DPT advances borings at the site.

This real-time data reduces level of uncertainty management as the results of the multiple sources of direct sensing information are simultaneously processed and displayed to both field and office team members simultaneously. The MIP/LIF and mobile laboratory data will also be used to select soil samples for off-site definitive laboratory analysis to confirm that a release has occurred by comparison of the laboratory analytical data to regulatory criteria. Details regarding the soil and groundwater sampling program are provided below.

17.1 SOIL SAMPLING DESIGN, LOCATIONS, AND RATIONALE

Soil data has not previously been collected at Site 2. Figure 17-1 provides the location of the environmental samples that are proposed to be collected within the Site 2 boundary. The Fire Fighter Training Area has been divided into a circular grid to guide the sampling locations and aid in the sample identification nomenclature. Initially, seven locations within the Fire Fighter Training Area pit will be screened from the ground surface to the water table (estimated to be 40 feet bgs) using the MIP/LIF to characterize the petroleum related compounds and/or chlorinated solvents that may have been released during the fire fighting training events and/or may have migrated due to subsequent precipitation events. Based on the real-time MIP/LIF data and mobile laboratory groundwater analytical results, up to six step out borings will be conducted approximately 3 feet outside of the Fire Fighter Training Area pit from the ground surface to the top of the water table (see Figure 17-1). These results will be used to select up to

seven additional sampling locations that will also be conducted from the ground surface to the top of the water table.

The real-time MIP/LIF results will be reviewed along with the mobile laboratory results for each groundwater sample to select up to 20 soil samples (surface and subsurface) for off-site laboratory analysis. Surface soil samples, if selected for laboratory analysis, will be collected from 0 to 2 feet bgs and will be analyzed for VOCs only. Subsurface soil samples will be based on the real-time MIP/LIF results and will be collected at intervals selected from 0.5 feet bgs to the top of the water table, which is estimated to be 40 feet bgs. The MIP/LIF DPT field screening results are real-time three dimensional semi-quantitative data that will be used to select samples (surface and subsurface soil and groundwater) for confirmation analysis by a fixed-base laboratory and to select the locations of monitoring wells that will be used to collect groundwater samples.

The MIP/LIF DPT three dimensional semi-quantitative data will be used to select 20 soil samples that represent high, medium, low and no responses by the MIP/LIF instrumentation. The confirmation soil samples will be selected in the field by the field operation lead to represent the high, medium, and low MIP/LIF instrument responses. The high, medium, and low MIP/LIF instrument responses will each consist of 30 percent of the 20 samples or 6 soil samples and the confirmation soil samples selected to represent no responses will be 10 percent of the 20 samples or 2 soil samples.

Soil samples collected for additional on-site field screening or off-site confirmation analysis will be field screened with an OVA. Up to 5 soil confirmation samples (described in Work Sheet 11) will be submitted to Katahdin for analysis of VOCs, SVOCs, pesticides, PCBs Florida Waste Oil Metals (arsenic, cadmium, chromium, and lead), and TRPH.

Based on MIP/LIF data, some sample locations shown on Figure 17-1 may be modified to a more strategic location or may not be performed altogether. The rationale in general would suggest that if contamination is not found in a boring, no additional borings will be advanced in the direction where contamination is not present. Sample locations may be moved to another location where more appropriate data may be obtained.

17.2 MIP/LIF DPT GROUNDWATER SAMPLING DESIGN, LOCATIONS, AND RATIONALE

Groundwater data has not previously been collected at Site 2. The DPT groundwater sampling will be supported by an on-site mobile laboratory that will be used to analyze groundwater samples collected by DPT. The DPT groundwater sample locations are the same as the soil sample locations shown on Figure 17-1. The groundwater samples will be collected from an interval between the top of the water table and 5 feet below the top of the water table encountered at the time of the field activities. The

groundwater samples will be analyzed for VOCs by a mobile laboratory (KB Labs) with the exception of replicate samples (described in Work Sheet 11) that will be submitted to an off-site fixed-base laboratory (Katahdin) as a QA measure (as described in Worksheet #18). The groundwater confirmation samples will be submitted for analysis of VOCs, SVOCs, pesticides, PCBs, Florida Waste Oil Metals (arsenic, cadmium, chromium, and lead), and TRPH.

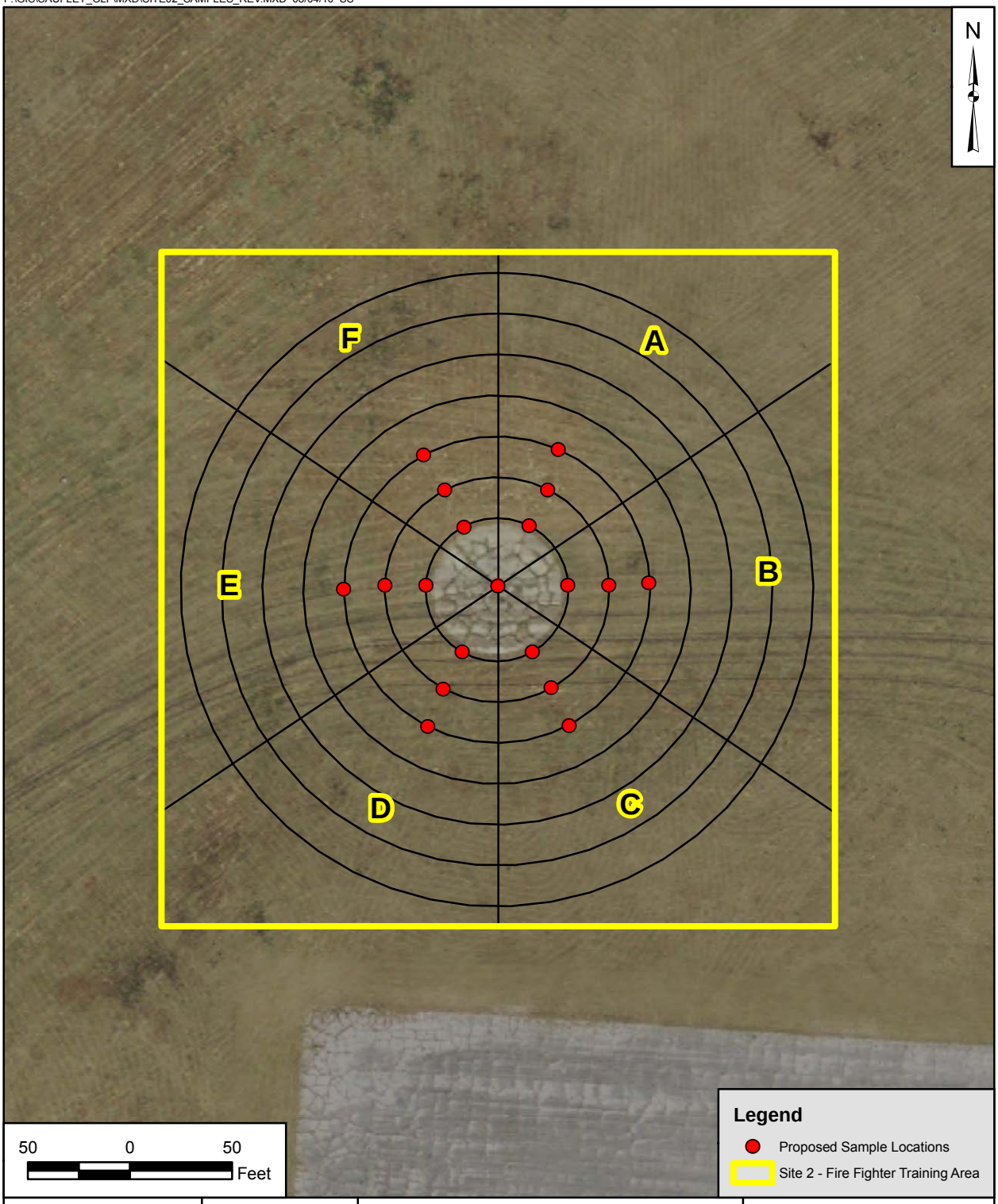
17.3 GROUNDWATER MONITORING WELL DESIGN, LOCATIONS, AND RATIONALE

After completion of the MIP/LIF DPT sampling program, the Project Team will evaluate MIP/DPT data from all areas at Site 2 for the purpose of designing, constructing and locating monitoring wells. The purpose of the monitoring wells (10 permanent shallow monitoring wells and three permanent deep monitoring wells) will be to provide confirmation of the plume extent, if found, that was identified during the sampling and analysis of groundwater samples collected at MIP/LIF DPT locations and to provide locations for future monitoring of natural attenuation processes and an appropriate long-term monitoring program. The final design and location details cannot be provided at this time, but is anticipated to consist of hydraulic upgradient, side gradient, and downgradient monitoring wells that are positioned based on the horizontal extent of the plume, if present, as determined by the MIP/LIF DPT soil and groundwater analytical results.

17.4 SAMPLING PROGRAM OPTIMIZATION

The Triad Approach allows for continual sample optimization through the review of near real-time data that allows for field decisions based on the obtained data. MIP/LIF data will be used to identify areas of high interest and low interest so that DPT sampling can be modified to collect more valued data in high interest areas and limit data collection in low interest areas. In addition, use of the on-site mobile laboratory will allow the Field Team to modify sample grids to eliminate samples in areas where contamination is not found to exist and to increase sampling density in areas where contamination is found. As a result, data produced is assured to be relevant and more complete, allowing for better decision making.

P:\GIS\SAUFLEY_OLF\MXD\SITE02_SAMPLES_REV.MXD 08/04/10 SS



DRAWN BY M. MAGUIRE	DATE 08/04/10	SITE 2 SOIL AND GROUND-WATER SAMPLE LOCATIONS SAUFLEY FIELD PENSACOLA, FLORIDA	CONTRACT NUMBER CTO JM30
CHECKED BY F. LESESNE	DATE 08/04/10		APPROVED BY ___ DATE ___
COST/SCHED AREA			APPROVED BY ___ DATE ___
SCALE AS NOTED			FIGURE NO. 17-1 REV 0

SAP Worksheet #18 -- Sampling Locations and Methods/SOP Requirements Table
(UFP-QAPP Manual Section 3.1.1)

The Triad Approach allows for continual sampling optimization. As a result, total sample numbers and depths are to be determined for MIP/LIF DPT soil and groundwater sampling efforts. Split samples will be analyzed in the fixed-base laboratory on a minimum of 5 percent of the VOC samples that are analyzed in the on-site mobile laboratory for each environmental medium (MIP/LIF DPT soil and groundwater). Field duplicates for each media will be collected on a minimum of 5 percent of the total samples for analysis. Sample designation protocol is as follows:

Surface Soil: Facility Identification, Site Identification, and Surface Soil Identification with sample sector letter and number, depth interval (bgs), month, and year.

Example: SF-2-SSA1-0-2-10/2010

Subsurface Soil: Facility Identification, Site Identification, and Subsurface Soil Identification with sample sector letter and number, depth interval (bgs), month, and year.

Example: SF-2-SBA1-0.5-2-10/2010

MIP/LIF DPT Groundwater: Facility Identification, Site Identification, and Boring Identification with sample sector letter and number, depth (screen) interval (bgs), month, and year.

Example: SF-2-GWA1-40-45-10/2010

New Monitoring Wells: Facility Identification, Site Identification, Area Identification, and Well Number with bottom of the screened interval (bgs), month, and year.

Example: SF-2-MW01-45-10/2010

Sampling Location/ Identification Number	Matrix	Synoptic Water Level	Well Depth (feet)	Analytical Group	Number of Field Duplicates	Sampling SOP Reference	Rationale for Sampling Location
Surface Soil Sample Locations							
SF-2-TBD-TBD-mm/yyyy	Soil	NA	NA	VOCs, SVOCs (Including Low Level SVOCs and PAHs by SIM), Pesticides, PCBs, Metals and TRPH.	5% minimum	CT-04, FC1000, FD1000, FS1000, FS3000 GH-1.5, SA-1.3, SA-6.1, SA-6.3, SA-7.1	Confirmation Sample

Note: Soil samples will be collected from the 20 proposed DPT locations within Site 2. Location numbers and sample depth will be based on the actual sample location selected for confirmation analysis.

Sampling Location/ Identification Number	Matrix	Synoptic Water Level	Well Depth[2](feet)	Analytical Group	Number of Field Duplicates	Sampling SOP Reference	Rationale for Sampling Location
DPT/MIP/LIF Sample Locations							
SF-2-GWA1TBD-TBD-mm/yyyy	Groundwater	NA	NA (5 foot interval below the water table)	VOCs (Screening)	5% (and 10% Off-site Confirmation Split Samples)	CT-04, FC1000, FD1000, FS1000, FS2212, FS2220, FT1000, FT1100, FT1200, FT1400, FT1500, FT1600, GH-1.5, SA-1.1, SA-1.2, SA-1.3, SA-2.5, SA-6.1, SA-6.3, SA-7.1	Plume Delineation
SF-2-GWA1TBD-TBD-mm/yyyy	Groundwater	NA	NA (5 foot interval below the water table)	VOCs (Screening)	5% (and 10% Off-site Confirmation Split Samples)	CT-04, FC1000, FD1000, FS1000, FS2000, FS2212, FS2220, FT1000, FT1100, FT1200, FT1400, FT1500, FT1600, GH-1.5, SA-1.1, SA-1.2, SA-1.3, SA-2.5, SA-6.1, SA-6.3, SA-7.1	Confirmation

Note: Groundwater samples for mobile laboratory analysis will be collected from the 20 proposed DPT locations within Site 2. Location numbers and sample depth will be based on the actual sample location selected for confirmation analysis.

Groundwater samples for fixed-base laboratory analysis will be collected from 5 of the 20 proposed DPT locations within Site 2. Location numbers and sample depth will be based on the actual sample location selected for confirmation analysis.

Sampling Location/ Identification Number	Matrix	Synoptic Water Level	Well Depth (feet)	Analytical Group	Number of Field Duplicates	Sampling SOP Reference	Rationale for Sampling Location
Monitoring Well Samples							
SF-2-MWTBD-TBD-mm/yyyy	Water	Yes	TBD	TBD	5% minimum	CT-04, FC1000, FD1000, FS1000, FS2212, FS2220, FT1000, FT1100, FT1200, FT1400, FT1500, FT1600, GH-1.2, GH-1.5, GH-2.5, GH-2.8, SA-1.1, SA-1.3, SA-2.5, SA-6.1, SA-6.3, SA-7.1	Monitoring
Note: Monitoring well locations are determined through the Triad Approach and sample location identifications will be determined according to location of installation at the time of installation.							

SAP Worksheet #19 -- Analytical SOP Requirements Table

(UFP-QAPP Manual Section 3.1.1)

Matrix	Analytical Group	Analytical and Preparation Method / SOP Reference[1]	Containers (number, size, and type)[2]	Sample Volume (units)	Preservation Requirements	Maximum Holding Time[(3)] (preparation / analysis)
Soil	VOCs	SW846 5035, 8260B / CA-202, CA-214	Three terra cores sample vials (5 gram [g] size)	5 g (each)	Sodium bisulfate / methanol/ water and freeze to -10 ° C	48 hours from sampling to preparation, 14 days to analysis
Groundwater and Aqueous QC Samples	VOCs	SW846 5030, 8260B / CA-202	Three 40 mL glass vials	40 mL	Hydrochloric acid (HCl) to pH<2; Cool to 0 to 6 °C; no headspace	14 days to analysis
Groundwater	VOCs Screening Level Data	SW-846 8260B KB SOP01VOC	Two 40 mL glass vials	5 mL	HCl to pH<2; Cool to 0 to 6 °C; no headspace	14 days to analysis
Soil	SVOCs (and Low Level SVOCs and PAHs by SIM)	SW846 3545A, 3550C, 8270D, 8270D SIM / CA-213, CA-226, CA-512, CA-526	One 8-ounce (oz) wide-mouth glass jar	30 g	Cool to 0 to 6 °C	14 days to extract / 40 days from extraction to analysis
Groundwater and Aqueous QC Samples	SVOCs (and Low Level SVOCs and PAHs by SIM)	SW846 3510C, 3520C, 8270D, 8270D SIM / CA-213, CA-226, CS-502	Two 1 L amber glass bottles	1 liter (L)	Cool to 0 to 6 °C	7 days to extract / 40 days from extraction to analysis
Soil	PCBs	SW846 3540C, 3545A, 3550C, 8082A / CA-329, CA-500, CA-524, CA-537	One 8 oz wide-mouth glass jar	30 g	Cool to 0 to 6 °C	14 days to extract / 40 days from extraction to analysis
Groundwater and Aqueous QC Samples	PCBs - low level modification	SW846 3510C, 3520C 8082A / CA-329, CA-515	Two 1 L amber glass bottles	1 L	Cool to 0 to 6 °C	7 days to extract / 40 days from extraction to analysis
Soil	Pesticides	SW846 3540C, 3545A, 3550C, 8081B / CA-302, CA-500, CA-524, CA-537	One 8 oz wide-mouth glass jar	30 g	Cool to 0 to 6 °C	14 days to extract / 40 days from extraction to analysis
Groundwater and Aqueous QC Samples	Pesticides - low level modification	SW846 3510C, 3520C 8081B / CA-302, CA-515	Two 1 L amber glass bottles	1 L	Cool to 0 to 6 °C	7 days to extract / 40 days from extraction to analysis
Soil	Metals (Including Mercury)	SW846 3050B, 6010C, 7471A / CA-605, CA-608, CA-611	One 4 oz wide-mouth glass jar	2 g / 0.3 g for mercury	Cool to 0 to 6 °C	6 months to analysis for all except mercury; mercury is 28 days to analysis.
Groundwater and Aqueous QC Samples	Metals (Including Mercury)	SW846 3010A, 6010C, 7470A / CA-604, CA-608, CA-615	One 1 L High Density Polyethylene bottle	200 mL	Nitric acid to pH <2; Cool to 0 to 6 °C	6 months to analysis for all except mercury; mercury is 28 days to analysis.
Soil	TRPH	FL-PRO/ CA-333	One 4 oz glass jar	30 g	Cool to 0 to 6 °C	7 days until extraction, 40 days to analysis

Matrix	Analytical Group	Analytical and Preparation Method / SOP Reference[1]	Containers (number, size, and type)[2]	Sample Volume (units)	Preservation Requirements	Maximum Holding Time[(3)] (preparation / analysis)
Groundwater and Aqueous QC Samples	TRPH	FL-PRO/ CA-333	1,000 mL	Two 1 L glass amber bottles	HCl to pH <2; Cool to 0 to 6 °C	7 days until extraction/40 days to analysis
IDW [4]	TCLP Organics	SW-846 1311, 3510, 5030B, 8260B, 8270D, 8081B, 8151A/ CA-202, CA-209, CA-226, CA-302, CA-305, CA-502, CA-510, CA-515, CA-516	Four 8 oz wide-mouth glass jars	400 g	Cool to 0 to 6 °C	48 hours for preparation; 14 days to analysis (VOCs only); 7 days to extraction; 40 days to analysis
	TCLP Inorganics	SW-846 1311, 3010, 6010C, 7470A / CA-510, CA-604, CA-608, CA-615	One 8 oz wide-mouth glass jar	100 g	Cool to 0 to 6 °C	180 days (28 days for mercury) to leach and analysis

Notes:

1 Laboratory SOPs are subject to revision and updates during duration of the project. The laboratory will use the most current revision of the SOP at the time of analysis.
2 Sample size is a minimum. The containers listed will be filled to compensate for any required re-analysis or re-extractions. For samples requiring Matrix Spike (MS)/Matrix Spike Duplicate (MSD), containers listed should be tripled.
3 Maximum holding time is calculated from the time the sample is collected to the time the sample is prepared/extracted.
4 IDW sample analyses are presented on this worksheet for the utilization of field personnel. QC information is not presented in any of the remaining worksheets as these samples are for waste disposal, not decision making purposes.

g = Gram
L = Liter
TCLP = Toxicity Characteristic Leaching Procedure

SAP Worksheet #20 -- Field Quality Control Sample Summary Table

(UFP-QAPP Manual Section 3.1.1)

Matrix	Analytical Group	Number of Sampling Locations	Number of Field Duplicates	Number of MS/MSDs[1]	Number of Field Blanks	Number of Equipment Blanks	Number of VOC Trip Blanks	Number of PT Samples	Total Number of Samples to Lab
Surface Soil	VOCs	20	2	1/1	0	0	2	0	24
	SVOCs / Low Level SVOCs and PAHs by SIM	20	2	1/1	0	0	NA	0	22
	Pesticides/PCBs	20	2	1/1	0	0	NA	0	22
	Metals	20	2	1/1	0	0	NA	0	22
	TRPH	20	2	1/1	0	0	NA	0	22
Groundwater (MIP/LIF DPT Samples)	VOCs (On-Site Screening)	20	2	2/2	0	0	0	0	22
Groundwater (MIP/LIF DPT Samples)	VOCs (Off-Site Confirmation)	2	0	0/0	0	0	0	0	2
Groundwater (Monitoring Wells)	VOCs	13	1	1/1	0	1	2	0	17
	SVOCs / Low Level SVOCs and PAHs by SIM	13	1	1/1	0	1	NA	0	15
	Pesticides/PCBs	13	1	1/1	0	1	NA	0	15
	Metals	13	1	1/1	0	1	NA	0	15
	TRPH	13	1	1/1	0	1	NA	0	15

Notes:

PT = Proficiency Testing

[1] Although MS/MSDs are not typically considered field QC samples, they are included here because location determination is often established in the field. The MS/MSDs are not included in the total number of samples sent to the laboratory.

SAP Worksheet #21 -- Project Sampling SOP References Table
(UFP-QAPP Manual Section 3.1.2)

REFERENCE NUMBER	TITLE, REVISION DATE AND/ OR NUMBER	ORIGINATING ORGANIZATION OF SAMPLING SOP[1]	EQUIPMENT TYPE	MODIFIED FOR PROJECT WORK? (Y/N)	COMMENTS
CT-04	Sample Nomenclature, Revision 2, March 2009	Tetra Tech	NA	Y	Sample names will follow the logic outlined in Worksheet #18. Contained in Appendix A.
CT-05	Database Records and Quality Assurance, Revision 2, January 29, 2001	Tetra Tech	NA	N	Contained in Appendix A.
FC 1000	Cleaning/Decontamination Procedures, December 2008	FDEP	Decontamination Equipment (scrub brushes, phosphate free detergent, de-ionized water)	N	Contained in Appendix A.
FD 1000	Documentation Procedures, December 2008	FDEP	Documentation of all sampling activities (log book, sampling logs, chain-of-custodies)	N	Contained in Appendix A.
FS 1000	General Sampling Procedures, December 2008	FDEP	NA	N	Contained in Appendix A.
FS 2212	Well Purging Techniques, December 2008	FDEP	Multi-parameter water quality meter, such as a Horiba U-22	N	Included in FDEP SOP FS 2200. Contained in Appendix A.
FS 2220	Groundwater Sampling Techniques, December 2008	FDEP	Multi-parameter water quality meter, such as a Horiba U-22	N	Contained in Appendix A.
FS 3000	Soil Sampling, December 2008	FDEP	Soil Sampling equipment	N	Contained in Appendix A.
FT 1000	Field Testing General, December 2008	FDEP	Multi-parameter water quality meter, such as a Horiba U-22	N	Contained in Appendix A.
FT 1100	Field pH, December 2008	FDEP	Multi-parameter water quality meter, such as a Horiba U-22	N	Contained in Appendix A.
FT 1200	Field Specific Conductance, December 2008	FDEP	Multi-parameter water quality meter, such as a Horiba U-22	N	Contained in Appendix A.
FT 1400	Field Temperature, December 2008	FDEP	Multi-parameter water quality meter, such as a Horiba U-22	N	Contained in Appendix A.

REFERENCE NUMBER	TITLE, REVISION DATE AND/ OR NUMBER	ORIGINATING ORGANIZATION OF SAMPLING SOP[1]	EQUIPMENT TYPE	MODIFIED FOR PROJECT WORK? (Y/N)	COMMENTS
FT 1500	Field DO, December 2008	FDEP	Multi-parameter water quality meter, such as a Horiba U-22	N	Contained in Appendix A.
FT 1600	Field Turbidity, December 2008	FDEP	Turbidity meter, such as LaMotte Model 2008, or similar	N	Contained in Appendix A.
GH-1.2	Evaluation of Existing Monitoring Wells and Water Level Measurement, Revision 2, September 2003	Tetra Tech	Electronic water level indicator	N	Contained in Appendix A.
GH-1.5	Borehole and Sample Logging, Revision 1, June 1999	Tetra Tech	NA	N	Contained in Appendix A.
HS-1.0	Utility Locating and Excavating Clearance, Revision 2, December 2003	Tetra Tech	Soil clearing equipment	N	Contained in Appendix A.
GH-2.5	Groundwater Contour Maps and Flow Determinations, Revision 1, June, 1999	Tetra Tech	NA	N	Contained in Appendix A.
GH-2.8	Groundwater Monitoring Well Installation, Revision 3, September, 2003	Tetra Tech	Health and safety equipment, well drilling and installation equipment, hydrogeologic equipment, drive point installation tools	N	Contained in Appendix A.
SA-6.1	Non-Radiological Sample Handling, Revision 3, February 2004	Tetra Tech	Sample Bottle Ware, Packaging Material, Shipping Materials	N	Contained in Appendix A.
SA-6.3	Field Documentation, Revision 3, March 2009	Tetra Tech	Field Logbook, Field Sample Forms, Boring Logs	N	Contained in Appendix A.
SA-7.1	Decontamination of Field Equipment, Revision 6, January 2009	Tetra Tech	Decontamination equipment, scrub brushes, 5-gallon buckets, spray bottles, phosphate-free detergent, DI water	N	Contained in Appendix A.
None	General Site Activities, 6/30/2008	Columbia Technologies	General Site Activities, Health and Safety Contingency Plan	N	Contained in Appendix A.

REFERENCE NUMBER	TITLE, REVISION DATE AND/ OR NUMBER	ORIGINATING ORGANIZATION OF SAMPLING SOP[1]	EQUIPMENT TYPE	MODIFIED FOR PROJECT WORK? (Y/N)	COMMENTS
None	Geoprobe Soil Sampling, 6/30/2009	Columbia Technologies	Geoprobe Soil Sampling	N	Contained in Appendix A.
None	Laser Induced Flourescense (LIF) Operation 2/27/2009	Columbia Technologies	LIF Operation	N	Contained in Appendix A.
None	Membrane Interface Probe Operation 7/27/2010	Columbia Technologies	MIP Operation	N	Contained in Appendix A.
None	Dakota Technologies UVOST Log Reference	Columbia Technologies	LIF Wave Length Reference	N	Contained in Appendix A.
None	Dakota Technologies General Operating Procedures	Columbia Technologies	LIF General Operating Procedures	N	Contained in Appendix A.
None	Standard Operating Procedures (SOP) Membrane Interface Probe System, 1/10/2007 Revision 3.0	Columbia Technologies	SOP Membrane Interface Probe System	N	Contained in Appendix A.

Notes:

[1] FDEP Field SOPs can be obtained at the following website: http://www.dep.state.fl.us/labs/bars/sas/sop/index.htm

SAP Worksheet #22 – Field Equipment Calibration, Maintenance, Testing, and Inspection Table

(UFP-QAPP Manual Section 3.1.2.4)

Field Equipment	Activity[1]	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[3]	Comments
Electric water level indicator and oil/water interface probe	Visual inspection and field checks as per manufacturer's guidance	Daily Once upon receiving from vendor	0.01 foot accuracy	Operator correction or replacement	Tetra Tech FOL or designee	GH-1.2, manufacturer's guidance manual	None
YSI 600 Series (or similar) Multi-parameter Water Quality Meter	Visual inspection and calibration/ verification	Daily Beginning and end of day	Manufacturer's guidance	Operator correction or replacement	Tetra Tech FOL or designee	FDEP FT 1000 through 1500 and manufacturer's guidance	None
LaMotte Model 2008 (or similar) Turbidity Meter	Visual inspection and calibration/ verification	Daily Beginning and end of day	RPD of ± 10% (6 measurements of 2 successive samples of a 20 NTU standard) Accuracy of ± 10% of 20 NUT (mean of the measured values must be 18 to 22 NTU)	Operator correction or replacement	Tetra Tech FOL or designee	FDEP FT 1600 and manufacturer's guidance	If an acceptable turbidity meter model is not used, submittal of an Alternate Test Procedure application is required

Notes:

[1] Activities may include calibration, verification, testing, maintenance, and/or inspection.
[2] Specify the appropriate reference letter or number from the Project Sampling SOP References table (Worksheet #21).
[3] FDEP Field SOPs can be obtained at the following website: http://www.dep.state.fl.us/labs/qa/sops.htm

SAP Worksheet #23 – Analytical SOP References Table

(UFP-QAPP Manual Section 3.2.1)

Laboratory SOP Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work?[1] (Y/N)
CA-202	Analysis of VOCs using Purge and Trap Gas Chromatography/Mass Spectrometer (GC/MS): SW-846 Method 8260, 04/10, Revision 11.	Definitive	Soil, Groundwater, and Aqueous QC Samples - VOCs	GC/MS	Katahdin	N
CA-213	Analysis of SVOCs using SW 846 Method 8270 – Modified For SIM, 04/10, Revision 8.	Definitive	Soil, Groundwater, and Aqueous QC Samples - Low Level SVOCs and PAHs	GC/MS	Katahdin	N
CA-214	Closed-System Purge-And-Trap And Extraction For Volatile Organics In Soil And Waste Samples using SW846 Method 5035, 09/08, Revision 5.	Definitive	Soil - VOCs	TEKMAR, ARCON, ENCON	Katahdin	N
CA-226	Analysis of SVOCs using Capillary Column GC/MS: SW-846 Method 8270D, 08/09, Revision 1.	Definitive	Soil, Groundwater, and Aqueous QC Samples - SVOCs	GC/MS	Katahdin	N
CA-302	Analysis of Pesticides using Gas Chromatography/Electron Capture Detector (GC/ECD): SW-846 Method 8081, 04/10, Revision 11.	Definitive	Soil, Groundwater, and Aqueous QC Samples - Pesticides	GC/ECD	Katahdin	N
CA-329	Analysis of PCBs as Total Aroclors using GC/ECD: SW-846 Method 8082, 04/10, Revision 11.	Definitive	Soil, Groundwater, and Aqueous QC Samples - PCBs	GC/ECD	Katahdin	Y – final extract volume of 2 mL
CA-333	Determination of Petroleum Range Organics using FDEP Method FL-PRO, 09/08, Revision 4.	Definitive	Soil, Groundwater, and Aqueous QC Samples - FL-PRO	GC/FID	Katahdin	N
CA-500	Preparation of Sediment/Soil Samples by Sonication using Method 3550 for Subsequent Pesticides/PCBs Analysis, 02/09, Revision 6.	Definitive	Soil - Pesticides and PCBs Extractions	Sonicator	Katahdin	N
CA-502	Preparation of Aqueous Samples for Extractable Semivolatile Analysis, 10/09, Revision 6.	Definitive	Groundwater and Aqueous QC Samples - SVOCs/ PAHs Extractions	Separatory Funnel, Continuous Liquid to Liquid Extraction (CLLE)	Katahdin	N
CA-512	Preparation of Sediment/Soil Samples by Sonication using Method 3550 for Subsequent Extractable Semivolatiles Analysis, 02/09, Revision 7.	Definitive	Soil - SVOCs/PAHs Extractions	Sonicator	Katahdin	N

Laboratory SOP Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work?[1] (Y/N)
CA-515	Preparation of Aqueous Samples for Pesticides/PCBs Analysis, 10/09, Revision 6.	Definitive	Groundwater and Aqueous QC Samples - Pesticides and PCBs Extractions	Separatory Funnel, CLLE	Katahdin	N
CA-524	Preparation of Sediment/Soil Samples by Soxhlet Extraction using Method 3540 for Pesticide/PCB Analysis, 08/09, Revision 6.	Definitive	Soil - Pesticides and PCBs Extractions	Soxhlet	Katahdin	N
CA-526	Preparation of Sediment/Soil Samples by Soxhlet Extraction using Method 3540 For Subsequent Extractable Semivolatile Analysis, 08/09, Revision 6.	Definitive	Soil - SVOCs/PAHs Extractions	Soxhlet	Katahdin	N
CA-537	Preparation of Sediment/Soil and Tissue Samples by Accelerated Solvent Extraction using Method 3545 for Subsequent Extractable Pesticide and PCB Analysis, 08/09, Revision 2.	Definitive	Soil - Pesticides and PCBs Extractions	Accelerate Solvent Extraction	Katahdin	N
CA-604	Acid Digestion of Aqueous Samples using USEPA Method 3010 for Inductively Coupled Plasma (ICP) Analysis of Total or Dissolved Metals, 04/10, Revision 5.	Definitive	Groundwater and Aqueous QC Samples - Metals Digestion	Block Digester	Katahdin	N
CA-605	Acid Digestion of Solid Samples using USEPA Method 3050 For Metals Analysis by ICP-Atomic Emission Spectroscopy (AES) and Graphite Furnace Atomic Absorption, 08/09, Revision 4.	Definitive	Soil - Metals Digestion	Block Digester	Katahdin	N
CA-608	Trace Metals Analysis by ICP-AES using USEPA Method 6010, 04/10, Revision 10.	Definitive	Soil, Groundwater, and Aqueous QC Samples - Metals	ICP-AES	Katahdin	N
CA-611	Digestion and Analysis of Solid Samples for Mercury using USEPA Method 7471, 04/10, Revision 7.	Definitive	Soil - Mercury	Cold Vapor Atomic Absorption (CVAA)	Katahdin	N
CA-615	Digestion and Analysis of Aqueous Samples for Mercury using USEPA Method 7470, 04/10, Revision 5.	Definitive	Groundwater and Aqueous QC Samples - Mercury	CVAA	Katahdin	N
SD-902	Sample Receipt and Internal Control, 08/09, Revision 8.	Definitive	Sample Receiving	NA	Katahdin	N
SD-903	Sample Disposal, 05/09, Revision 4.	Definitive	Sample Receiving	NA	Katahdin	N
KBSOP01VOC	Analytical SOP Number 1, Determination of VOCs by Purge and Trap GC/MS Method 8260B (Revision 4, July 2008)	Screening	Groundwater - VOCs	GC/MS	KB Labs	N

SAP Worksheet #24 -- Analytical Instrument Calibration Table

(UFP-QAPP Manual Section 3.2.2)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference[1]
GC/MS VOCs	Initial Calibration (ICAL) – A minimum 5-point calibration is required	Calibrate the instrument when it is received, after a major change (source cleaning, new column, change in GC run parameters), or if the daily calibration fails.	The average Response Factors (RFs) for System Performance Check Compounds (SPCCs) are 1,1,2,2-tetrachloroethane and chlorobenzene must be ≥0.30 and chloromethane, 1,1-dichloroethane, and bromoform must be ≥0.10. The Percent Relative Standard Deviations (%RSDs) for RFs of Calibration Check Compounds (CCCs) must be ≤ 30%, and the %RSDs must be ≤ 15% for all target analytes. If not met: Option 1) Linear least squares regression: Linear Regression Correlation Coefficient (r) must be ≥ 0.995; or Option 2) Non-linear regression: coefficient of determination (r^2) must be ≥ 0.990 (6 points are required for second order).	Repeat calibration if criterion is not met.	Analyst, Supervisor	CA-202
	Retention Time (RT) Window Position Establishment	Once per ICAL for each analyte and surrogate.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial continuing calibration verification is used.	NA.	Analyst, Supervisor	
	Evaluation of Relative Retention Times (RRTs)	With each sample.	RRT of each target analyte must be within ± 0.006 RRT units.	Correct problem, then rerun ICAL.	Analyst, Supervisor	
	Initial Calibration Verification (ICV) – Second Source	Once after each ICAL prior to sample analysis.	Percent Recovery (%R) must be within 80-120% for all target analytes.	Correct problem and verify ICV. Reanalyze ICV and/or ICAL as appropriate.	Analyst, Supervisor	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference[1]
	Continuing Calibration Verification (CCV)	Analyze a standard at the beginning of each 12-hour shift after a bromofluorobenzene (BFB) tune.	Percent Difference or Percent Drift (%D) must be ≤ 20% for all target analytes and surrogates. RFs for SPCCs must be ≥0.10 and ≥0.30 (compounds as listed above in ICAL block).	Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since last acceptable CCV.	Analyst, Supervisor	
	BFB Tune	Prior to ICAL and at the beginning of each 12-hour analytical sequence.	Criteria listed in Table 4 of Katahdin SOP CA-202. No samples may be analyzed without a valid tune.	Retune and/or clean source.	Analyst, Supervisor	
GC/MS VOCs screening by mobile lab	ICAL – A minimum of a 5-point calibration is prepared for all target analytes	Prior to any sample analysis.	The %RSD of target analyte RFs must be ≤ 15%. Minimum mean RFs of SPCCs as listed in SW-846 8260B must be met during the ICAL. The %RSDs of CCC RFs during ICAL must be ≤ 30%.	Correct problem and repeat ICAL. Do not analyze samples until ICAL passes criteria.	Analyst	KB Labs SOP01VOC
	CCV – A midlevel standard run every 12 hours prepared from separate source from calibration standards	Daily before sample analysis and every 12 hours of analysis time.	RF criteria for SPCCs the same as during ICAL. RF of CCCs must be ≤ 20 %D from ICAL.	Rerun CCV. Then rerun ICAL, if necessary.	Analyst	
	BFB Tune	Prior to ICAL and at the beginning of each 12-hour analytical sequence.	Criteria listed in SW-846 8260B.	Retune and/or clean source.	Analyst	
GC/MS SVOCs (including SVOCs and PAHs by SIM)	Breakdown Check (DDT only)	At the beginning of each 12-hour analytical sequence.	The degradation must be ≤ 20% for DDT to verify inertness of the injection port.	Correct the problem then repeat breakdown check. No samples shall be run until degradation is ≤20% for DDT.	Analyst, Supervisor	CA-213, CA-226

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference[1]
	ICAL – A minimum 5-point calibration is required	Calibrate the instrument when it is received, after a major change (source cleaning, new column, change in GC run parameters), or if the daily calibration fails.	Average RF SPCCs must be ≥0.050 (≥0.010 for SIM); %RSD for RFs for CCCs must be ≤30%; and the %RSD must be ≤ 15% for all other compounds. If not met: Option 1) r must be ≥ 0.995, or Option 2) r^2 must be ≥ 0.99 (minimum of 6 points required for second order). For low-level SVOCs and PAHs, the %RSD must be ≤ 20%, or meet one of the above options.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards.	Analyst, Supervisor	
	ICV – Second Source	Once after each ICAL prior to sample analysis	%D must be ≤ 20% for all target analytes.	Correct problem and verify second source standard. Reanalyze ICV and/or ICAL as appropriate.	Analyst, Supervisor	
	RT Window Position Establishment	Once per ICAL for each analyte and surrogate.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	Analyst, Supervisor	
	Evaluation of RRTs	With each sample.	RRT of each target analyte must be within ± 0.006 RRT units.	Correct problem, then rerun ICAL.	Analyst, Supervisor	
	CCV	Analyze a standard at the beginning of each 12-hour shift after a decafluorotriphenyl-phosphine (DFTPP) tune.	%D must be ≤ 20% for all target analytes and surrogates. SPCCs RFs must be >0.050 (≥0.010 for SIM).	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst, Supervisor	
	DFTPP Tune	Prior to initial calibration and at the beginning of each 12-hour analytical sequence.	Criteria listed in Section 7.4 of Katahdin SOP CA-213 and Section 7.4 of CA-226. No samples may be analyzed without a valid tune.	Retune and/or clean source.	Analyst, Supervisor	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference[1]
GC/ECD PCBs	ICAL – A minimum 5- point calibration curve is run for Aroclor 1016 and 1260 and a single-point reference for all other Aroclors. If an Aroclor other than 1016/1260 is identified in any sample by peak pattern, then the sample is re-analyzed with a full calibration curve for that Aroclor	Instrument receipt, major instrument change, when CCV does not meet criteria.	6-point calibration of Aroclors 1016/1260, 1242, 1248, and 1254 – One of the options below: Option 1: RSD for each analyte must be ≤ 20%; Option 2: r must be ≥ 0.995; Option 3: r^2 must be ≥ 0.99 (6 points shall be used for second order). Midpoint calibration of Aroclors 1221 and 1232; if these target analytes are detected, a 6-point calibration is performed and the samples are reanalyzed.	Repeat ICAL and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data.	Analyst, Supervisor	CA-329
	ICV – Second Source	Once after each ICAL and prior to sample analysis.	%D of all target analytes must be ≤ 20%.	Identify source of problem, correct, repeat calibration, rerun samples.	Analyst, Supervisor	
	CCV	Once after each ICAL and at the beginning and end of each run sequence and every 10 samples.	%D of all target analytes must be ≤ 20%.	Identify source of problem, correct, repeat calibration, rerun samples.	Analyst, Supervisor	
GC/ECD Pesticides	ICAL – A 6-point calibration of individual pesticides, with a mid-point calibration of chlordane and toxaphene	Upon instrument receipt, major instrument change, and when the CCV does not meet criteria.	The RSD for RFs for each target analyte must be ≤ 20%, or r must be ≥ 0.995, or r^2 must be ≥ 0.99 (minimum of 6 points required for second order).	Correct problem and repeat ICAL. If single point calibration analyte chlordane and toxaphene is identified in analysis of sample, 6-point calibration run of identified compound with reanalysis of sample.	Analyst, Supervisor	CA-302

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference[1]
	ICV – Second Source	Once after each initial calibration prior to sample analysis.	%Rs of target analytes must be within 80-120% of accepted value.	Identify source of problem, correct problem, repeat ICAL, rerun samples. Do not analyze samples until ICV passes criteria	Analyst, Supervisor	
	CCV	Once after each initial calibration and at the beginning and end of each run sequence and every 10 samples.	%Ds of target analytes must be ≤20%.	Identify source of problem, correct problem, repeat ICAL, Reanalyze all samples analyzed since last successful CCV.	Analyst, Supervisor	
	Breakdown Check (pesticides only)	Perform daily prior to sample analysis.	The degradation must be ≤ 15% for both DDT and Endrin to verify the inertness of the injection port.	Correct the problem then repeat breakdown check. No samples shall be run until degradation is ≤15% for both DDT and Endrin.	Analyst, Supervisor	
ICP-AES Metals (Except Mercury)	ICAL – One point calibration for each element	Daily prior to sample analysis, and if continuing QC fails.	None; only one high standard and a calibration blank must be analyzed. If more than one calibration standard is used, r must be ≥ 0.995.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards.	Analyst, Supervisor	CA-608
	ICV – Second Source	Once after each ICAL and prior to sample analysis.	%R must be within 90-110% of true value.	Do not use results for failing elements, unless ICV >110% and sample result < reporting limit.	Analyst, Supervisor	
	CCV	At the beginning and end of each run sequence and every 10 samples.	%R must be within 90-110% of true value.	Check problem, recalibrate and reanalyze any samples not bracketed by passing CCVs.	Analyst, Supervisor	
	Initial Calibration Blank (ICB)	Before beginning a sample sequence.	No analyte detected > LOD.	Correct the problem, then re-prepare and reanalyze.	Analyst, Supervisor	
	Continuing Calibration Blank (CCB)	After the initial CCV, after every 10 samples, and at the end of the sequence	No analyte detected > LOD.	Correct the problem, then re-prepare and reanalyze calibration blank and all affected samples.	Analyst, Supervisor	
	Low-Level Calibration Check Standard (if using 1-point ICAL)	At beginning and end of run.	%R must be within 80%-120% of true value.	Do not use results for failing elements, unless LOQ recovery > upper limit and sample result < LOQ/reporting limit.	Analyst, Supervisor	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference[1]
	Interference Check Standards (ICS) – ICS A and ICSA B)	Daily, before sample injections.	ICS A recoveries must be less than the absolute value of the LOD and ICSA B %Rs must be within 80-120% of the true value.	Correct the problem, then re-prepare checks and reanalyze all affected samples.	Analyst, Supervisor	
CVAA Mercury	ICAL – A 6-point calibration curve is analyzed	Daily prior to sample analysis, and if continuing QC fails.	The RSD for RFs must be ≤ 20% or r must be ≥ 0.995.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards	Analyst, Supervisor	CA-611, CA-615
	ICB	Before beginning a sample sequence.	No mercury detected > LOD.	Correct problem, re-prepare, and reanalyze.	Analyst, Supervisor	
	ICV – Second Source	Once after each ICAL and prior to sample analysis	%R must be within 90-110% of the true value.	Correct problem and repeat calibration.	Analyst, Supervisor	
	CCB	After each CCV, after every 10 samples, and at the end of the sequence	No mercury detected > LOD.	Investigate source of contamination, rerun any samples not bracketed by passing blanks	Analyst, Supervisor	
	CCV	CCV-at beginning and end of each run sequence and every 10 samples.	%R must be within 80-120% of the true value.	Check problem, recalibrate and reanalyze any samples not bracketed by passing CCVs.	Analyst, Supervisor	
GC/FID TRPH	ICAL – A minimum of a 5-point calibration is prepared for all target analytes	Upon instrument receipt, major instrument change, or when the CCV does not meet criteria.	The RSD for RFs for each target analyte must be ≤ 20%, or r must be ≥ 0.995.	Correct problem then repeat ICAL. No samples may be run until ICAL has passed.	Analyst, Supervisor	CA-333
	ICV – Second Source	Following ICAL, prior to the analysis of samples.	The %R must be within 80-120% of true value.	Correct problem and verify ICV. If that fails, correct problem and repeat ICAL. No samples may be run until ICV has been verified.	Analyst, Supervisor	
	CCV	At the beginning of a sequence and after every 12 hours or 10 samples (whichever comes first), then at the end of the sequence.	The %R must be within 75-125% of true value.	Correct problem and rerun CCV. If that fails, repeat ICAL and reanalyze all samples analyzed since the last successful CCV. If the CCV fails high, report samples that are less than the LOQ.	Analyst, Supervisor	

[1] Laboratory SOPs are subject to revision and updates during duration of the project, the laboratory will use the most current revision of the SOP at the time of analysis.

SAP Worksheet #25 -- Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

(UFP-QAPP Manual Section 3.2.3)

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
GC/MS	Check pressure, gas supply and vacuum daily. Bake out trap and column, manual tune if BFB not in criteria, change septa as needed, cut column as needed, change trap as needed, clean MS source as needed. Other maintenance specified in Laboratory Equipment Maintenance SOP.	VOCs	Ion source, injector liner, column, column flow, purge lines, purge flow, trap.	Prior to ICAL and/or as necessary.	Acceptable ICAL or CCV.	Correct the problem and repeat ICAL or CCV.	Analyst, Supervisor	CA-202
GC/MS	Check pressure, gas supply, and vacuum daily. Bake out column, manual tune if DFTPP not in criteria, change septa as needed, cut column as needed, clean MS source as needed. Other maintenance specified in Laboratory Equipment Maintenance SOP.	SVOCs (including Low Level SVOCs and PAHs by SIM)	Ion source, injector liner, column, column flow.	Prior to ICAL and/or as necessary.	Acceptable ICAL or CCV.	Correct the problem and repeat ICAL or CCV.	Analyst, Supervisor	CA-213, CA-226
GC/ECD	Check pressure and gas supply daily. Change septa and/or liner as needed, replace or cut column as needed. Other maintenance specified in Laboratory Equipment Maintenance SOP.	PCBs/ Pesticides	Injector liner, septa, column, column flow.	Prior to ICAL and/or as necessary.	Acceptable ICAL or CCV.	Correct the problem and repeat ICAL or CCV.	Analyst, Supervisor	CA-302, CA-329

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
ICP-AES	Clean sample path, check pump tubing, argon level, vacuum and waste container daily. Clean source as needed. Other maintenance specified in Laboratory Equipment Maintenance SOP.	Metals (except mercury)	Pump, pump tubing, vacuum source, waste container.	Prior to ICAL and as necessary.	Acceptable ICAL or CCV.	Correct the problem and repeat ICAL or CCV.	Analyst, Supervisor	CA-608
CVAA	Replace peristaltic pump tubing, replace mercury lamp, replace drying tube, clean optical cell and/or clean liquid/gas separator as needed. Other maintenance specified in Laboratory Equipment Maintenance SOP.	Mercury	Tubing, sample probe, optical cell, waste container.	Prior to ICAL and as necessary.	Acceptable ICAL or CCV.	Correct the problem and repeat ICAL or CCV.	Analyst, Supervisor	CA-611, CA-615
GC-FID	Check pressure and gas supply daily. Change septa and/or GC injector glass liner as needed. Replace or cut GC column as needed. Other maintenance specified in Laboratory Equipment Maintenance SOP.	TRPH FL-PRO	Injector liner, septa, column, column flow.	Prior to ICAL and as necessary.	Acceptable ICAL or CCV.	Correct the problem and repeat ICAL or CCV.	Analyst, Supervisor	CA-333

SAP Worksheet #26 -- Sample Handling System
(UFP-QAPP Manual Appendix B)

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): Tetra Tech FOL or designee / Tetra Tech
Sample Packaging (Personnel/Organization): Tetra Tech FOL or designee / Tetra Tech
Coordination of Shipment (Personnel/Organization): Tetra Tech FOL or designee / Tetra Tech
Type of Shipment/Carrier: Federal Express
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): Sample Custodians / Katahdin and KB Labs
Sample Custody and Storage (Personnel/Organization): Sample Custodians/ Katahdin and KB Labs
Sample Preparation (Personnel/Organization): Extraction Laboratory, Metals Preparation Laboratory / Katahdin and KB Labs
Sample Determinative Analysis (Personnel/Organization): Gas Chromatography Laboratory, GC/MS Laboratory, Metals Laboratory / Katahdin; and Mobile GC/MS Laboratory, KB Laboratory
SAMPLE ARCHIVING
Field Sample Storage (Number of days from sample collection): 60 days from receipt
Sample Extract/Digestate Storage (Number of days from extraction/digestion): 3 months from sample digestion/extraction
Biological Sample Storage (Number of days from sample collection): N/A
SAMPLE DISPOSAL
Personnel/Organization: Sample Custodians / Katahdin and KB Labs

SAP Worksheet #27 -- Sample Custody Requirements Table
(UFP-QAPP Manual Section 3.3.3)

27.1 SAMPLE NOMENCLATURE, SAMPLE COLLECTION DOCUMENTATION, HANDLING, AND TRACKING PROCEDURES

The following sections outline the procedures that will be used to document project activities and sample collection, handling, tracking, and custody procedures during the investigation. All forms must be filled in as completely as possible.

27.1.1 Sample Nomenclature

Refer to Worksheet #18 for how the samples will be labeled. Also, refer to Worksheet #20 for how the field QA/QC samples will be labeled.

Sample nomenclature will be conducted in general accordance with the procedures outlined in Tetra Tech SOP CT-04 (Sample Nomenclature). Sample nomenclature put forth for this field event has been selected based on historical usage. The sample nomenclature for each tracking number includes the site being investigated, sample media identifier, and sample location number. The standard sample matrix and type codes used for this field event are as follows: Duplicate samples will be submitted to the laboratory as blind duplicates. The QA/QC type codes used for this field event are as follows: TB for Trip Blanks and RB for rinsate blanks. Field QC blanks will be labeled sequentially followed by the date (i.e., TB-20101213, FB-20101214, etc.). Samples to be used for MS and MSDs will be labeled MS/MSD on the container label and noted on the chain-of-custody, as required in the laboratory QA Plan; however, MS/MSD will not be part of the unique sample identifier in order to maintain consistency with the project database. Additional information regarding protocol for sample labeling is contained in Tetra Tech SOP SA-6.3 (see Appendix A).

27.1.2 Sample Collection Documentation

Documentation of field observations will be recorded in a field logbook and/or field log sheets including sample collection logs, boring logs, VOC screening logs, and monitoring well construction logs. Field logbooks utilized on this project will consist of a bound, water-resistant logbook. All pages of the logbook will be numbered sequentially and observations will be recorded with indelible ink.

Field sample log sheets will be used to document sample collection details and other observations and activities will be recorded in the field logbook. Instrument calibration logs will be used to record the daily instrument calibration. Example field forms are included in Appendix A.

For sampling and field activities, the following types of information will be recorded in the field log as appropriate:

- Site name and location
- Date and time of logbook entries
- Personnel and their affiliations
- Weather conditions
- Activities involved with the sampling
- Subcontractor activity summary
- Site observations including site entry and exit times
- Site sketches made on-site
- Visitor names, affiliations, arrival and departure times
- Health and safety issues, including PPE

27.1.3 Sample Handling and Tracking System

Following sample collection into the appropriate bottle ware, all samples will be immediately placed on ice in a cooler. The glass sample containers will be enclosed in bubble-wrap in order to protect the bottle ware during shipment. The cooler will be secured using strapping tape along with a signed custody seal. Sample coolers will be delivered to a local courier location for priority overnight delivery to the selected laboratory for analysis. Samples will be preserved as appropriate based on the analytical method. The laboratories will provide pre-preserved sample containers for sample collection. Samples will be maintained at 0 to 6 °C until delivery to the laboratory. Proper custody procedures will be followed throughout all phases of sample collection and handling.

After collection, each sample will be maintained in the sampler's custody until formally transferred to another party (e.g., Federal Express). For all samples collected, chain-of-custody forms will document the date and time of sample collection, the sampler's name, and the names of all others who subsequently held custody of the sample. Specifications for chemical analyses will also be documented on the chain-of-custody form. Tetra Tech SOP SA-6.3 (Field Documentation) provides further details on the chain-of-custody procedure, which is provided in Appendix A.

These subsections outline the procedures that will be used by field and laboratory personnel to document project activities and sample collection procedures during this SI. All forms must be filled in as completely as possible.

Sample handling requirements are described in Worksheet #26. Tetra Tech personnel will collect the samples. The samplers will take care not to contaminate samples through improper handling. Samples will be sealed in appropriate containers, packaged by Tetra Tech personnel and placed into sealed coolers under chain-of-custody in accordance with the applicable SOP (See Worksheet #21). Samples to be analyzed for VOCs will be accompanied by a VOC trip blank. All coolers will contain a temperature blank. Samples will be transferred under chain-of-custody to a courier as described below. Once received by the laboratory, receipt will be documented on the chain-of-custody form and the samples will be checked in. The samples will remain under chain-of-custody throughout the analysis period to ensure their integrity is preserved. Details are provided below.

Samples to be delivered to the laboratory(s) will be made by a public courier (i.e., Federal Express). After samples have been collected, they will be sent to the laboratory(s) within 24 hours. Under no circumstances will sample holding times be exceeded.

27.2 FIELD SAMPLE CUSTODY PROCEDURES

Chain-of-custody protocols will be used throughout sample handling to establish the evidentiary integrity of sample containers. These protocols will be used to demonstrate that the samples were handled and transferred in a manner that would eliminate possible tampering. Samples for the laboratory will be packaged and shipped in accordance with Tetra Tech SOP SA-6.1 (see Appendix A).

A sample is under custody if:

- The sample is in the physical possession of an authorized person.
- The sample is in view of an authorized person after being in his/her possession.
- The sample is placed in a secure area by an authorized person after being in his/her possession.
- The sample is in a secure area, restricted to authorized personnel only.

Custody documentation is designed to provide documentation of preparation, handling, storage, and shipping of all samples collected. A multi-part form is used with each page of the form signed and dated by the recipient of a sample or portion of sample. The person releasing the sample and the person receiving the sample each will retain a copy of the form each time a sample transfer occurs.

Integrity of the samples collected during the site investigation will be the responsibility of identified persons from the time the samples are collected until the samples, or their derived data, are incorporated into the final report.

The Tetra Tech FOL is responsible for the care and custody of the samples collected until they are delivered to the laboratory or are entrusted to a carrier. When transferring samples, the individuals relinquishing and receiving them will sign, date, and note the time on the chain-of-custody form. This record documents the sample custody transfer from the sampler to the laboratory, often through another person or agency (common carrier). Upon arrival at the laboratory, internal sample custody procedures will be followed as defined in the Laboratory SOPs included in Appendix B.

27.3 LABORATORY CHAIN OF CUSTODY – KATAHDIN AND KB LABS

Laboratory sample custody procedures (receipt of samples, archiving, and disposal) will be used according to Katahdin and KB Labs Laboratory SOPs (see Appendix B). Coolers are received and checked for proper temperature and preservation. A sample cooler receipt form will be filled out to note conditions and any discrepancies. The chain of custody will be checked against the sample containers for correctness. Samples will be logged into the Laboratory Information Management System and given a unique log number that can be tracked through processing. The Laboratory PM will notify the Tetra Tech FOL of any problems on the same day that the issue is identified.

SAP Worksheet #28 -- Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix	**Soil, Groundwater, and Aqueous QC Samples**
Analytical Group	**VOCs**
Analytical Method/SOP Reference	**SW-846 8260B/ CA-202**

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Method Blank	One is performed for each batch of up to 20 samples.	No target analyte concentrations may be > ½ the LOQ, except common laboratory contaminants methylene chloride, acetone, and 2-butanone may be present, but must be < LOQ.	If blank results are above ½/LOQ (or > LOQ for common contaminants), sample results which are < LOQ or > 10X the blank contamination concentration may be reported without corrective action. Otherwise, re-analyze all associated samples.	Analyst, Supervisor, Data Validator	Contamination/ Bias	Same as Method/SOP QC Acceptance Limits.
Laboratory Control Sample (LCS)	One is performed for each batch of up to 20 samples.	%Rs must meet the limits provided in the Katahdin QC Limits table provided in Appendix B.	Correct problem, then re-prepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. Contact Client if samples cannot be reanalyzed within hold time.	Analyst, Supervisor, Data Validator	Accuracy	Same as Method/SOP QC Acceptance Limits.

Matrix	**Soil, Groundwater, and Aqueous QC Samples**
Analytical Group	**VOCs**
Analytical Method/SOP Reference	**SW-846 8260B/ CA-202**

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
MS/MSD	One set is performed for each batch of up to 20 samples.	%Rs should meet the limits provided in the Katahdin QC Limits table provided in Appendix B. The RPD between MS and MSD should be ≤ 30%.	Failure to meet the control limits shall be discussed in the case narrative. If both the LCS and MS/MSD are unacceptable, all associated samples must be re-analyzed.	Analyst, Supervisor, Data Validator	Accuracy / Precision	Same as Method/SOP QC Acceptance Limits.
Surrogates	4 per sample: Dibromofluoromethane 1,2-dichloroethane-d_4 Toluene-d_8 Bromofluorobenzene	%Rs must meet the limits provided in the Katahdin QC Limits table provided in Appendix B.	Re-analyze affected samples if volume is available.	Analyst, Supervisor, Data Validator	Accuracy / Bias	Same as Method/SOP QC Acceptance Limits.
Internal Standards (IS)	4 per sample- Pentafluorobenzene 1,4-Difluorobenzene Chlorobenzene-d_5 1,4-Dichlorobenzene-d_4	RTs must be within ± 30 seconds and the response areas must be within -50% to +100% of the last ICAL midpoint standard for each IS.	Re-analyze affected samples if volume is available.	Analyst, Supervisor, Data Validator	Accuracy / Bias	Same as Method/SOP QC Acceptance Limits.

Note that limits are updated periodically and may change from the issuance of the final SAP to the time data validation is performed. The limits used for validation will be the limits that are current at the time of analysis.

Matrix	**Soil, Groundwater, and Aqueous QC Samples**
Analytical Group	**SVOCs, Low Level SVOCs and PAHs by SIM**
Analytical Method / SOP Reference	**SW-846 8270D, 8270D SIM/ CA-213, CA-226**

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Method Blank	One is performed for each batch of up to 20 samples of the same matrix.	No target analytes > ½ the LOQ.	If blank results are above ½ LOQ, sample results which are < LOQ or > 10X the blank contamination concentration may be reported without corrective action. Otherwise, re-extract all associated samples.	Analyst, Supervisor, Data Validator	Contamination / Bias	Same as Method/SOP QC Acceptance Limits.
LCS	One is performed for each batch of up to 20 samples of the same matrix.	%Rs must meet the limits provided in the Katahdin QC Limits table provided in Appendix B.	Correct problem, then re-prepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available Contact client if samples cannot be reanalyzed within hold time.	Analyst, Supervisor, Data Validator	Accuracy / Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	**Soil, Groundwater, and Aqueous QC Samples**
Analytical Group	**SVOCs, Low Level SVOCs and PAHs by SIM**
Analytical Method / SOP Reference	**SW-846 8270D, 8270D SIM/ CA-213, CA-226**

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
MS/MSD	One set is performed for each batch of up to 20 samples of the same matrix.	%Rs should meet the limits provided in the Katahdin QC Limits table provided in Appendix B. The RPD between MS and MSD should be ≤ 30%.	Failure to meet the control limits shall be discussed in the case narrative. If both the LCS and MS/MSD are unacceptable, all associated samples must be re-analyzed.	Analyst, Supervisor, Data Validator	Accuracy / Precision	Same as Method/SOP QC Acceptance Limits.
Surrogates	6 per sample: 2-Fluorophenol Phenol-d_6 Nitrobenzene-d_5 2-Fluorobiphenyl 2,4,6-Tribromophenol Terphenyl-d_{14} For low-level SVOCs/PAHs, 3 surrogates per sample: 2-Methylnaphthalene-d_{10} Fluorene-d_{10} Pyrene-d_{10}	%Rs must meet the limits provided in the Katahdin QC Limits table provided in Appendix B.	Field samples having one or more surrogate recoveries above the control limits will not require corrective action if no associated target analytes (acid analytes with acid surrogates, etc.) are detected > LOQ. Otherwise, affected samples must be reanalyzed.	Analyst, Supervisor, Data Validator	Accuracy / Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	**Soil, Groundwater, and Aqueous QC Samples**
Analytical Group	**SVOCs, Low Level SVOCs and PAHs by SIM**
Analytical Method / SOP Reference	**SW-846 8270D, 8270D SIM/ CA-213, CA-226**

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
IS	6 per sample: 1,4-Dichlorobenzene-d_4 Naphthalene-d_8 Acenaphthene-d_{10} Phenanthrene-d_{10} Chrysene-d_{12} Perylene-d_{12}	RTs for ISs must be within ±30 seconds and the response areas must be within -50% to +100% of the last ICAL midpoint standard for each IS.	Re-analyze affected samples if volume is available.	Analyst, Supervisor, Data Validator	Accuracy / Bias	Same as Method/SOP QC Acceptance Limits.

Note that limits are updated periodically and may change from the issuance of the final SAP to the time data validation is performed. The limits used for validation will be the limits that are current at the time of analysis.

Matrix	**Soil, Groundwater, and Aqueous QC Samples**
Analytical Group	**PCBs**
Analytical Method / SOP Reference	**SW-846 8082A/ CA-329**

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Method Blank	One is performed for each batch of up to 20 samples of the same matrix.	No target analyte concentrations may be > ½ the LOQ.	If blank results are above ½ LOQ, sample results which are < LOQ or > 10X the blank contamination concentration may be reported without corrective action. Otherwise, re-extract all associated samples.	Analyst, Supervisor, Data Validator	Contamination / Bias	Same as Method/SOP QC Acceptance Limits.
LCS	One is performed for each batch of up to 20 samples of the same matrix.	%Rs must meet the limits provided in the Katahdin QC Limits table provided in Appendix B.	Correct problem, then re-prepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available Contact Client if samples cannot be re-prepared within hold time.	Analyst, Supervisor, Data Validator	Accuracy	Same as Method/SOP QC Acceptance Limits.
MS/MSD	One set is performed for each batch of up to 20 samples of the same matrix.	%Rs should meet the limits provided in the Katahdin QC Limits table provided in Appendix B. The RPD between MS and MSD should be ≤ 30%.	Failure to meet the control limits shall be discussed in the case narrative. If both the LCS and MS/MSD are unacceptable, all associated samples must be re-analyzed.	Analyst, Supervisor, Data Validator	Precision / Accuracy	Same as Method/SOP QC Acceptance Limits.

Matrix	**Soil, Groundwater, and Aqueous QC Samples**
Analytical Group	**PCBs**
Analytical Method / SOP Reference	**SW-846 8082A/ CA-329**

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Surrogates	2 per sample: Decachlorobiphenyl Tetrachloro-m-xylene	%Rs must meet the limits provided in the Katahdin QC Limits table provided in Appendix B.	For QC and field samples, correct problem then re-prepare and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary. Contact client if samples cannot be re-prepared within hold time.	Analyst, Supervisor, Data Validator	Accuracy	Same as Method/SOP QC Acceptance Limits.
Second Column Confirmation	All positive results must be confirmed.	Results between primary and second column must be RPD ≤ 40%. The higher of the two results will be reported unless matrix interference is apparent.	None. Apply qualifier if RPD >40% and discuss in the case narrative.	Analyst, Supervisor, Data Validator	Precision	Same as Method/SOP QC Acceptance Limits.

Note that limits are updated periodically and may change from the issuance of the final SAP to the time data validation is performed. The limits used for validation will be the limits that are current at the time of analysis.

Matrix	**Soil, Groundwater, and Aqueous QC Samples**
Analytical Group	**Pesticides**
Analytical Method / SOP Reference	**SW-846 8081B/ CA-302**

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Method Blank	One is performed for each batch of up to 20 samples of the same matrix.	No target analytes > ½ the LOQ.	If blank results are above ½ LOQ, sample results which are < LOQ or > 10X the blank contamination concentration may be reported without corrective action. Otherwise, re-extract all associated samples.	Analyst, Supervisor, Data Validator	Contamination / Bias	Same as Method/SOP QC Acceptance Limits.
LCS	One is performed for each batch of up to 20 samples of the same matrix.	%Rs must meet the limits provided in the Katahdin QC Limits table provided in Appendix B.	Correct problem, then re-prepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available (see full explanation in Appendix B). Contact Client if samples cannot be re-prepped within hold time.	Analyst, Supervisor, Data Validator	Accuracy	Same as Method/SOP QC Acceptance Limits.
MS/MSD	One set is performed for each batch of up to 20 samples of the same matrix.	%Rs should meet the limits provided in the Katahdin QC Limits table provided in Appendix B. The RPD between MS and MSD should be ≤ 30%.	Failure to meet the control limits shall be discussed in the case narrative. If both the LCS and MS/MSD are unacceptable, all associated samples must be re-analyzed.	Analyst, Supervisor, Data Validator	Precision / Accuracy	Same as Method/SOP QC Acceptance Limits.

Matrix	**Soil, Groundwater, and Aqueous QC Samples**
Analytical Group	**Pesticides**
Analytical Method / SOP Reference	**SW-846 8081B/ CA-302**

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Surrogates	2 per sample: Decachlorobiphenyl Tetrachloro-m-xylene	%Rs must meet the limits provided in the Katahdin QC Limits table provided in Appendix B.	For QC and field samples, correct problem then re-prepare and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary. Contact client if samples cannot be re-prepped within hold time.	Analyst, Supervisor, Data Validator	Accuracy	Same as Method/SOP QC Acceptance Limits.
Second Column Confirmation	All positive results must be confirmed.	Results between primary and second column must be RPD ≤ 40%. The higher of the two results will be reported unless matrix interference is apparent.	None. Apply qualifier if RPD >40% and discuss in the case narrative.	Analyst, Supervisor, Data Validator	Precision	Same as Method/SOP QC Acceptance Limits.

Note that limits are updated periodically and may change from the issuance of the final SAP to the time data validation is performed. The limits used for validation will be the limits that are current at the time of analysis.

Matrix	**Soil, Groundwater, and Aqueous QC Samples**
Analytical Group	**Metals (Including Mercury)**
Analytical Method/SOP Reference	**SW-846 6010C, 7470A, 7471A/ CA-608, CA-611, CA-615**

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Method Blank	One is performed for each batch of up to 20 samples of the same matrix.	No target analytes > the LOQ.	If blank results are above LOQ, sample results that are < LOQ or > 10X the blank contamination concentration may be reported without corrective action. Otherwise, re-extract all associated samples.	Analyst, Supervisor, Data Validator	Contamination / Bias	Same as Method/SOP QC Acceptance Limits.
LCS	One is performed for each batch of up to 20 samples of the same matrix.	%R must be within 80-120% of the true value.	Failure of any element will necessitate a re-digestion/re-analysis of all associated samples for that element.	Analyst, Supervisor, Data Validator	Accuracy	Same as Method/SOP QC Acceptance Limits.
Sample Duplicate	One sample duplicate is performed for each batch of 20 samples of the same matrix.	The RPD should be ≤20%.	Failure to meet the control limit shall be discussed in the case narrative, and elements will be flagged accordingly.	Analyst, Supervisor, Data Validator	Precision	Same as Method/SOP QC Acceptance Limits.
MS	One MS is performed for each batch of up to 20 samples of the same matrix.	%Rs should be within 80 to 120%, if sample < 4x spike added.	Failure to meet the control limits shall be discussed in the case narrative, and elements will be flagged accordingly.	Analyst, Supervisor, Data Validator	Accuracy	Same as Method/SOP QC Acceptance Limits.

Matrix	**Soil, Groundwater, and Aqueous QC Samples**
Analytical Group	**Metals (Including Mercury)**
Analytical Method/SOP Reference	**SW-846 6010C, 7470A, 7471A/ CA-608, CA-611, CA-615**

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Serial Dilution (SD) (does not apply to mercury)	One SD (5x) is performed for each batch of 20 samples of the same matrix.	If original sample result is at least 50x the instrument detection limit, the SD must agree within ± 10% D of the original result.	Failure to meet the control limit shall be discussed in the case narrative, and elements will be flagged accordingly.	Analyst, Supervisor, Data Validator	Precision	Same as Method/SOP QC Acceptance Limits.
Post-Digestion Spike (does not apply to mercury)	For any element that fails in the MS where the native sample concentration was <4x the spike amount.	%R must be within 75 to 125% of the true value.	Discussed in the case narrative	Analyst, Laboratory Supervisor, Data Validator	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.

Note that limits are updated periodically and may change from the issuance of the final SAP to the time data validation is performed. The limits used for validation will be the limits that are current at the time of analysis.

Matrix	**Soil, Groundwater, and Aqueous QC Samples**
Analytical Group	**TRPH**
Analytical Method/SOP Reference	**FL-PRO/ CA-333**

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Method Blank	One per preparatory batch of 20 or fewer samples.	The target analyte must be ≤ ½ LOQ	Investigate source of contamination. Evaluate the samples and associated QC: i.e., if the blank results are >LOQ, then report sample results which are <LOQ or >10X the blank concentration. Otherwise, re-prepare a blank and samples >LOQ and <10X the blank.	Analyst, Supervisor, Data Validator	Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
Surrogate	Two per sample.	o-Terphenyl 30-140% %R. 2-Fluorobiphenyl 50-150 %R.	If surrogates %Rs are high and sample is <LOQ, then no corrective action is taken. If surrogates %Rs are low, then the affected samples are re-extracted and reanalyzed.	Analyst, Supervisor, Data Validator	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
LCS	One per preparatory batch of 20 or fewer samples.	%Rs must meet the limits provided in the Katahdin QC Limits table provided in Appendix B.	If an MS/MSD was performed and is acceptable, then narrate. If the LCS recovery is high, but the sample results are <LOQ, then narrate. Otherwise, re-extract blank and affected sample batch.	Analyst, Supervisor, Data Validator	Accuracy/ Bias/ Precision	Same as Method/SOP QC Acceptance Limits.
MS/MSD	One per preparatory batch of 20 or fewer samples per matrix.	%Rs must meet the limits provided in the Katahdin QC Limits table provided in Appendix B. The RPD between MS and MSD should be ≤ 30%.	Evaluate the samples and associated QC and if the LCS results are acceptable, then narrate. If both the LCS and MS/MSD are unacceptable, then re-prepare the samples and QC.	Analyst, Supervisor, Data Validator	Accuracy/ Bias/ Precision	Same as Method/SOP QC Acceptance Limits.

Note that limits are updated periodically and may change from the issuance of the final SAP to the time data validation is performed. The limits used for validation will be the limits that are current at the time of analysis.

Matrix	**Groundwater**
Analytical Group	**VOCs, Screening Mobile Lab**
Analytical Method/ SOP Reference	**SW-846 8260B KBSOP01VOC**

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Method Blank	One per daily analysis batch.	No analytes > 1/2 of the LOQ.	Bake out purge and trap system, change adsorbent trap. Re-prepare and reanalyze method blank and associated samples.	Analyst	Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
Surrogate	Four per sample: 1,4-dichlorobenzene 1,2-dichloroethane-d_4 Toluene-d_8 BFB	Should be within limits established by lab or method.	Reanalyze sample. If one or more still remain outside criteria, then recalibrate and/or remake surrogate solution.	Analyst	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
MS/MSD	One per 20 samples of similar matrix.	Should be within limits established by lab.	Check LCS to see if matrix effects apply.	Analyst	Accuracy/ Bias/ Precision	Same as Method/SOP QC Acceptance Limits.
LCS	One per daily analysis batch.	Must be within limits established by lab.	Re-prepare and reanalyze LCS. Reanalyze associated samples.	Analyst	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
IS	Three per sample- Fluorobenzene Chlorobenzene-d_5 1,4-dichlorobezene-d_4	RTs for ISs must be within ± 30 seconds and the response areas must be within -50% to +100% of the last calibration check.	Reanalyze sample. If one or more still remain outside criteria, recalibrate.	Analyst	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.

SAP Worksheet #29 – Project Documents and Records Table

(UFP-QAPP Manual Section 3.5.1)

Document	Where Maintained
Field Documents Field Logbook Field Sample Forms Chain of Custody Records Air Bills Sampling Instrument Calibration Logs Sampling Notes Photographs FTMR Forms This SAP HASP	Field documents will be maintained in the project file located in the Tetra Tech Pittsburgh office.
Laboratory Documents Sample receipt, custody, and tracking record Equipment calibration logs Sample preparation logs Analysis Run logs Corrective action forms Reported field sample results Reported results for standards, QC checks, and QC samples Extraction/clean-up records Raw data	Laboratory documents will be included in the hardcopy and portable documents format deliverables from the laboratory. Laboratory data deliverables will be maintained in the Tetra Tech Pittsburgh project file and in long-term data package storage at a third-party professional document storage firm. Electronic data results will be maintained in a database on a password protected Structured Query Language (SQL) server.
Assessment Findings Field Sampling Audit Checklist (if conducted) Analytical Audit Checklist (if conducted) Data Validation Memoranda (includes tabulated data summary forms)	All assessment documents will be maintained in the Tetra Tech Pittsburgh office.
Reports SI Report	All reports will be stored in hardcopy in the Tetra Tech Pittsburgh project file and electronically in the server library.

SAP Worksheet #30 -- Analytical Services Table

(UFP-QAPP Manual Section 3.5.2.3)

Matrix	Analytical Group	Sample Locations/ID Numbers	Analytical Method	Data Package Turnaround Time	Laboratory / Organization (name and address, contact person and telephone number)	Backup Laboratory/ Organization (name and address, contact person and telephone number)
Soil, Groundwater, and Aqueous QC Samples	VOCs	See Worksheet #18	SW-846 8260B	21 calendar days	Katahdin Analytical Services, Inc. 600 Technology Way Scarborough, Maine 04074 Ms. Kate Zaleski kzaleski@katahdinlab.com (207) 874-2400	NA
	SVOCs (including low level SVOCs and PAHs by SIM)		SW-846 8270D/ 8270D SIM			
	Pesticides		SW-846 8081B			
	PCBs		SW-846 8082A			
	Metals		SW-846 6010C, 7470A, and 7471A			
	TRPH		FL-PRO			
Groundwater	VOCs – mobile laboratory screening	See Worksheet #18	SW-846 8260B	Results within 24 hours	KB Labs, Inc. 25132 SW 1st Ave Newberry, Florida 32669 Todd Romero toddr@kbmobilelabs.com (352) 472-5830 (352) 472-5832 (fax)	NA

SAP Worksheet #31 -- Planned Project Assessments Table

(UFP-QAPP Manual Section 4.1.1)

Assessment Type	**Frequency**	**Internal or External**	**Organization Performing Assessment**	**Person(s) Responsible for Performing Assessment** (title and organizational affiliation)	**Person(s) Responsible for Responding to Assessment Findings** (title and organizational affiliation)	**Person(s) Responsible for Identifying and Implementing Corrective Action** (title and organizational affiliation)	**Person(s) Responsible for Monitoring Effectiveness of Corrective Action** (title and organizational affiliation)
Laboratory System Audit[1]	Every 2 years	External	DoD ELAP Accrediting Body	DoD ELAP Accrediting Body Auditor	Laboratory QAM or Laboratory Manager, Katahdin	Laboratory QAM or Laboratory Manager, Katahdin	Laboratory QAM or Laboratory Manager, Katahdin

[1] Katahdin has successfully completed the laboratory assessment process required as part of the DoD Quality Systems Manual Version 4.1 under the DoD ELAP by a recognized Accrediting Body. The DoD ELAP accreditation letter for Katahdin is included in Appendix B. KB Labs, Inc. (mobile laboratory) is NELAP accredited in the state of Florida. The NELAP accreditation letter for KB Labs is included in Appendix B.

SAP Worksheet #32 -- Assessment Findings and Corrective Action Responses Table

(UFP-QAPP Manual Section 4.1.2)

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (name, title, organization)	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (name, title, organization)	Timeframe for Response
Laboratory System Audit	Written audit report	Leslie Dimond, Laboratory QAM, Katahdin	Specified by DoD ELAP Accrediting Body	Letter	DoD ELAP Accrediting Body	Specified by DoD ELAP Accrediting Body

SAP Worksheet #33 -- QA Management Reports Table

(UFP QAPP Manual Section 4.2)

Type of Report	Frequency (daily, weekly monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (title and organizational affiliation)	Report Recipient(s) (title and organizational affiliation)
Data Validation Report	Per sample delivery group	Within 3 weeks of receipt of laboratory data package	DVM or designee, Tetra Tech	PM and project file, Tetra Tech
Major Analysis Problem Identification (Internal Tetra Tech Memorandum)	When persistent analysis problems are detected by Tetra Tech that may impact data usability	Immediately upon detection of problem (on the same day)	CLEAN QAM, Tetra Tech	PM, CLEAN QAM, Program Manager, and project file, Tetra Tech
Project Monthly Progress Report	Monthly for duration of project	Monthly	PM, Tetra Tech	Navy RPM, Navy; CLEAN QAM, Program Manager, and project file, Tetra Tech
Laboratory QA Report	When significant plan deviations result from unanticipated circumstances	Immediately upon detection of problem (on the same day)	Laboratory PM, Katahdin	PM and project file, Tetra Tech

SAP Worksheet #34 -- Verification (Step I) Process Table

(UFP-QAPP Manual Section 5.2.1)

Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
Chain-of-custody forms	The Tetra Tech FOL or designee will review and sign the chain-of-custody form to verify that all samples listed are included in the shipment to the laboratory and the sample information is accurate. The forms will be signed by the sampler and a copy will be retained for the project file, the Tetra Tech PM, and the Tetra Tech Data Validators. See Tetra Tech SOP SA-6.3.	Internal	Sampler and FOL, Tetra Tech
	The Laboratory Sample Custodian will review the sample shipment for completeness, integrity, and sign accepting the shipment. The Tetra Tech Data Validators will check that the chain-of-custody form was signed and dated by the Tetra Tech FOL or designee relinquishing the samples and also by the Laboratory Sample Custodian receiving the samples for analyses.	Internal/ External	1 - Laboratory Sample Custodian, Katahdin and KB Labs 2 - Data Validators, Tetra Tech
SAP Sample Tables/ Chain-of-Custody Forms	Verify that all proposed samples listed in the SAP tables have been collected.	Internal	FOL or designee, Tetra Tech
Sample Log Sheets	Verify that information recorded in the log sheets is accurate and complete.	Internal	FOL or designee, Tetra Tech
Sample Coordinates	Verify that actual sample locations are correct and in accordance with the SAP proposed locations. Document any discrepancies in the final report.	Internal	PM, FOL, or designee, Tetra Tech
SAP/ Field Logs/ Analytical Data Packages	Ensure that all sampling SOPs were followed. Verify that deviations have been documented and MPCs have been achieved. Particular attention should be given to verify that samples were correctly identified, that sampling location coordinates are accurate, and that documentation establishes an unbroken trail of documented chain-of-custody from sample collection to report generation. Verify that the correct sampling and analytical methods/SOPs were applied. Verify that the sampling plan was implemented and carried out as written and that any deviations are documented.	Internal	PM or designee, Tetra Tech
SAP/ Laboratory SOPs/ Raw Data/ Applicable Control Limits Tables	Ensure that all laboratory SOPs were followed. Verify that the correct analytical methods/SOPs were applied. Establish that all method QC samples were analyzed and in control as listed in the analytical SOPs. If method QA is not in control, the Laboratory QAM will contact the Tetra Tech PM via telephone or e-mail for guidance prior to report preparation.	Internal	Laboratory QAM, Katahdin and Data specialist, KB Labs
SAP/ Chain-of-Custody Forms	Check that field QC samples listed in Worksheet #20 were collected as required.	Internal	FOL or designee, Tetra Tech
Analytical Data Packages	All analytical data packages will be verified internally for completeness by the laboratory performing the work. The Laboratory QAM will sign the case narrative for each data package.	Internal	Laboratory QAM, Katahdin and Data specialist, KB Labs

SAP Worksheet #35 -- Validation (Steps IIa and IIb) Process Table

(UFP-QAPP Manual Section 5.2.2) (Figure 37 UFP-QAPP Manual) (Table 9 UFP-QAPP Manual)

Step IIa / IIb	Validation Input	Description	Responsible for Validation (name, organization)
IIa	Chain-of-Custody Forms	Custody - Ensure that the custody and integrity of the samples was maintained from collection to analysis and the custody records are complete and any deviations are recorded. Review that the samples were shipped and stored at the required temperature and sample pH for chemically-preserved samples meet the requirements listed in Worksheet #19. Ensure that the analyses were performed within the holding times listed in Worksheet #19.	Project Chemist or Data Validators, Tetra Tech
IIa/IIb	SAP/ Laboratory Data Packages/ Electronic Data Deliverables (EDDs)	Accuracy - Ensure that the laboratory QC samples listed in Worksheet #28 were analyzed and that the MPCs listed in Worksheet #12 were met for all field samples and QC analyses. Check that specified field QC samples were collected and analyzed and that the analytical QC criteria set up for this project were met.	Project Chemist or Data Validators, Tetra Tech
		Precision - Check the field sampling precision by calculating the RPD for field duplicate samples. Check the laboratory precision by reviewing the RPD or percent difference values from laboratory duplicate analyses; MS/MSDs; and LCS/laboratory control sample duplicate (LCSD), if available. Ensure compliance with the methods and project MPCs accuracy goals listed in Worksheet #12.	
		Representativeness - Check that the laboratory recorded the temperature at sample receipt and the pH of the chemically preserved samples to ensure sample integrity from sample collection to analysis.	
		Completeness - Review the chain-of-custody forms generated in the field to ensure that the required analytical samples have been collected, appropriate sample identifications have been used, and correct analytical methods have been applied. The Tetra Tech Data Validator will verify that elements of the data package required for validations are present, and if not, the laboratory will be contacted and the missing information will be requested. Validation will be performed as per Worksheet #36. Check that all data have been transferred correctly and completely to the final SQL database.	
IIb	SAP/ Laboratory Data Packages/ EDDs	Sensitivity - Ensure that the project LOQs listed in Worksheet #15 were achieved.	Project Chemist or Data Validators, Tetra Tech
		PALs - Discuss the impact on reported LDLs due to matrix interferences or sample dilutions performed because of the high concentration of one or	

Step IIa / IIb	Validation Input	Description	Responsible for Validation (name, organization)
		more other contaminants, on the other target analytes reported as non-detected. Document this usability issue and inform the Tetra Tech PM. Review and add PALs to the laboratory EDDs. Flag samples and notify the Tetra Tech PM of samples that exceed PALs listed in Worksheet #15.	
		QA/QC - Ensure that all QC samples specified in the SAP were collected and analyzed and that the associated results were within prescribed SAP acceptance limits. Ensure that QC samples and standards prescribed in analytical SOPs were analyzed and within the prescribed control limits. If any significant QC deviations occur, the Laboratory QAM shall have contacted the Tetra Tech PM.	
		Deviations - Summarize deviations from methods, procedures, or contracts in the Data Validation Report. Determine the impact of any deviation from sampling or analytical methods and SOPs requirements and matrix interferences effect on the analytical results. Qualify data results based on method or QC deviation and explain all the data qualifications. Print a copy of the project database qualified data depicting data qualifiers and data qualifiers codes that summarize the reason for data qualifications. Determine if the data met the MPCs and determine the impact of any deviations on the technical usability of the data.	

SAP Worksheet #36 -- Analytical Data Validation (Steps IIa and IIb) Summary Table

(UFP-QAPP Manual Section 5.2.2.1)

Step IIa / IIb	Matrix	Analytical Group	Validation Criteria	Data Validator (title and organizational affiliation)
IIa and IIb	Soil, Groundwater, and Aqueous QC Samples	VOCs, SVOCs (Including Low Level SVOCs and PAHs by SIM), Pesticides, PCBs, and TRPH by FL-PRO	100% Limited data * validation will be performed. SW-846 8260B, 8270D, 8270D SIM, 8081B, 8082A, and FL-PRO method specific criteria and those criteria listed in Worksheets #12, #15, #24, and #28. If not included in Worksheets #12, #15, #24, or #28, then the logic outlined in USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review EPA-540/R-99-008, October 1999 will be used to apply qualifiers to data.	Data Validation Specialist, Tetra Tech
IIa and IIb	Soil, Groundwater, and Aqueous QC Samples	Metals (Including Mercury)	100% Limited* data validation will be performed. SW-846 6010C, 7470A, and 7471A method specific criteria and those listed in Worksheets #12, #15, #24, and #28. If not included in Worksheets #12, #15, #24 or #28, then the logic outlined in USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review EPA 540-R-04-004, October 2004 will be used to apply qualifiers to data.	Data Validation Specialist, Tetra Tech

* Limited data validation. Limits the data review to specific review parameters (Data Completeness/Data Verification, Holding times, Calibrations, Blank Contamination, and Detection Limits) to determine gross deficiencies only. The limited data validation is best expressed as a review to preclude the possibility of false negatives and to eliminate false positives. Raw data are not evaluated and sample result verification is not conducted. A formal data validation report is prepared.

Mobile laboratory VOCs data reports will not be validated.

SAP Worksheet #37 -- Usability Assessment
(UFP-QAPP Manual Section 5.2.3)

Data Usability Assessment

The usability of the data directly affects whether project objectives can be achieved. At a minimum, the following characteristics will be evaluated. The results of these evaluations will be included in the project report. The characteristics will be evaluated for multiple concentration levels if the evaluator determines that this is necessary. To the extent required by the type of data being reviewed, the assessors will consult with other technically competent individuals to render sound technical assessments of these DQI characteristics:

Completeness

For each matrix that was scheduled to be sampled, the Tetra Tech FOL acting on behalf of the Project Team will prepare a table listing planned samples/analyses to collected samples/analyses. If deviations from the scheduled sample collection or analyses are identified the Tetra Tech PM and Project Risk Assessor will determine whether the deviations compromise the ability to meet project objectives. If they do, the Tetra Tech PM will consult with the Navy RPM and other Project Team members, as necessary (determined by the Navy RPM), to develop appropriate corrective actions.

Precision

The Tetra Tech Project Chemist acting on behalf of the Project Team will determine whether precision goals for field duplicates and laboratory duplicates were met. This will be accomplished by comparing duplicate results to precision goals identified in Worksheets #12 and #28. This will also include a comparison of field and laboratory precision with the expectation that field duplicate results will be no less precise than laboratory duplicate results. If the goals are not met, or data have been flagged as estimated (J qualifier), limitations on the use of the data will be described in the project report.

Accuracy

The Tetra Tech Project Chemist acting on behalf of the Project Team will determine whether the accuracy/bias goals were met for project data. This will be accomplished by comparing percent recoveries of LCS, LCSD, MS, MSD, and surrogate compounds to accuracy goals identified in Worksheet #28. This assessment will include an evaluation of field and laboratory contamination; instrument calibration variability; and analyte recoveries for surrogates, MS, and laboratory control samples. If the goals are not met, limitations on the use of the data will be described in the project report. Bias of the qualified results and a description of the impact of identified non-compliances on a specific data package or on the overall project data will be described in the project report.

Representativeness

A Tetra Tech Project Scientist identified by the Tetra Tech PM and acting on behalf of the Project Team will determine whether the data are adequately representative of intended populations, both spatially and temporally. This will be accomplished by verifying that samples were collected and processed for analysis in accordance with the SAP, by reviewing spatial and temporal data variations, and by comparing these characteristics to expectations. The usability report will describe the representativeness of the data for each matrix and analytical fraction. This will not require quantitative comparisons unless professional judgment of the project scientist indicates that a quantitative analysis is required.

Data Usability Assessment

Comparability

The Tetra Tech Project Chemist acting on behalf of the Project Team will determine whether the data generated under this project are sufficiently comparable to historical site data generated by different methods and for samples collected using different procedures and under different site conditions. This will be accomplished by comparing overall precision and bias among data sets for each matrix and analytical fraction. This will not require quantitative comparisons unless professional judgment of the Tetra Tech Project Chemist indicates that such quantitative analysis is required.

Sensitivity

The Tetra Tech Project Chemist acting on behalf of the Project Team will determine whether project sensitivity goals listed in Worksheet #15 are achieved. The overall sensitivity and quantitation limits from multiple data sets for each matrix and analysis will be compared. If sensitivity goals are not achieved, the limitations on the data will be described. The Tetra Tech Project Chemist will enlist the help of the Tetra Tech Risk Assessor to evaluate deviations from planned sensitivity goals.

Project Assumptions and Data Outliers

The Tetra Tech PM and designated team members will evaluate whether project assumptions are valid. This will typically be a qualitative evaluation but may be supported by quantitative evaluations. The type of evaluation depends on the assumption being tested. Quantitative assumptions include assumptions related to data distributions (e.g., normal versus log-normal) and estimates of data variability. Statistical tests for outliers will be conducted using standard statistical techniques appropriate for this task. Potential outliers will be removed if a review of the associated data indicates that the results have an assignable cause the renders them inconsistent with the rest of the data. During this evaluation, the team will consider whether outliers could be indications of unanticipated site conditions. Consideration will be given to whether outliers represent an unanticipated site condition.

Describe the evaluative procedures used to assess overall measurement error associated with the project:

After completion of the data validation, the data and data quality will be reviewed to determine whether sufficient data of acceptable quality are available for decision making. In addition to the evaluations described above, a series of inspections and statistical analyses will be performed to estimate these characteristics. The statistical evaluations will include simple summary statistics for target analytes, such as maximum concentration, minimum concentration, number of samples exhibiting non-detected results, number of samples exhibiting positive results, and the proportion of samples with detected and non-detected results. The Project Team members identified by the Tetra Tech PM will assess whether the data collectively support the attainment of project objectives. They will consider whether any missing or rejected data have compromised the ability to make decisions or to make the decisions with the desired level of confidence. The data will be evaluated to determine whether missing or rejected data can be compensated by other data. Although rejected data will generally not be used, there may be reason to use them in a weight of evidence argument, especially when they supplement data that have not been rejected. If rejected data are used, their use will be supported by technically defensible rationales.

For statistical comparisons and mathematical manipulations, non-detected values will be represented by a concentration equal to one-half the sample-specific reporting limit. Duplicate

Data Usability Assessment
results (original and duplicate) will not be averaged for the purpose of representing the range of concentrations. However, the average of the original and duplicate samples will be used to represent the concentration at a particular sampled location.
Identify the personnel responsible for performing the usability assessment: The Tetra Tech PM, Project Chemist, FOL, and Project Scientist will be responsible for conducting the listed data usability assessments. The data usability assessment will be reviewed with the Navy RPM, the USEPA RPM, and the FDEP RPM. If deficiencies affecting the attainment of project objectives are identified, the review will take place either in a face to face meeting or a teleconference depending on the extent of identified deficiencies. If no significant deficiencies are identified, the data usability assessment will simply be documented in the project report and reviewed during the normal document review cycle.
Describe the documentation that will be generated during usability assessment and how usability assessment results will be presented so that they identify trends, relationships (correlations), and anomalies: The data will be presented in tabular format, including data qualifications such as estimation (J, UJ) or rejection (R). Written documentation will support the non-compliance estimated or rejected data results. The project report will identify and describe the data usability limitations and suggest re-sampling or other corrective actions, if necessary.

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APPENDIX A

FIELD SOPS AND FIELD DATA SHEETS

TABLE OF CONTENTS

TABLE OF CONTENTS (Continued)

FIGURES

1.0 PURPOSE

The purpose of this document is to establish standard procedures and technical guidance on borehole and sample logging.

2.0 SCOPE

These procedures provide descriptions of the standard techniques for borehole and sample logging. These techniques shall be used for each boring logged to provide consistent descriptions of subsurface lithology. While experience is the only method to develop confidence and accuracy in the description of soil and rock, the field geologist/engineer can do a good job of classification by careful, thoughtful observation and by being consistent throughout the classification procedure.

3.0 GLOSSARY

None.

4.0 RESPONSIBILITIES

Site Geologist. Responsible for supervising all boring activities and assuring that each borehole is completely logged. If more than one rig is being used on site, the Site Geologist must make sure that each field geologist is properly trained in logging procedures. A brief review or training session may be necessary prior to the start up of the field program and/or upon completion of the first boring.

5.0 PROCEDURES

The classification of soil and rocks is one of the most important jobs of the field geologist/engineer. To maintain a consistent flow of information, it is imperative that the field geologist/engineer understand and accurately use the field classification system described in this SOP. This identification is based on visual examination and manual tests.

5.1 Materials Needed

When logging soil and rock samples, the geologist or engineer may be equipped with the following:

- Rock hammer
- Knife
- Camera
- Dilute hydrochloric acid (HCl)
- Ruler (marked in tenths and hundredths of feet)
- Hand Lens

5.2 Classification of Soils

All data shall be written directly on the boring log (Figure 1) or in a field notebook if more space is needed. Details on filling out the boring log are discussed in Section 5.5.

FIGURE 1
BORING LOG (EXAMPLE)

BORING LOG

Page ___ of ___

PROJECT NAME: _____ BORING NUMBER: _____
PROJECT NUMBER: _____ DATE: _____
DRILLING COMPANY: _____ GEOLOGIST: _____
DRILLING RIG: _____ DRILLER: _____

Sample No. and Type or RQD	Depth (Ft.) or Run No.	Blows / 6" or RQD (%)	Sample Recovery / Sample Length	Lithology Change (Depth/Ft.) or Screened Interval	MATERIAL DESCRIPTION: Soil Density/ Consistency or Rock Hardness	MATERIAL DESCRIPTION: Color	MATERIAL DESCRIPTION: Material Classification	USCS *	Remarks	PID/FID Reading (ppm): Sample	PID/FID Reading (ppm): Sampler BZ	PID/FID Reading (ppm): Borehole**	PID/FID Reading (ppm): Driller BZ**

* When rock coring, enter rock brokeness.

** Include monitor reading in 6 foot intervals @ borehole. Increase reading frequency if elevated response read.

Remarks: _____

Drilling Area Background (ppm): ☐

Converted to Well: Yes _____ No _____ Well I.D. #: _____

FIGURE 1 (CONTINUED)

SOIL TERMS

UNIFIED SOIL CLASSIFICATION (USCS)

COARSE-GRAINED SOILS
More Than Half of Material is LARGER Than No. 200 Sieve Size

FIELD IDENTIFICATION PROCEDURES (Excluding Particles Larger Than 3 Inches and Basing Fractions on Estimated Weights)			GROUP SYMBOL	TYPICAL NAMES
GRAVELS 50% or more of coarse fraction > No. 4 sieve	CLEAN GRAVELS (Low % Fines)	Wide range in grain size and substantial amounts of all intermediate particle sizes.	GW	Well graded gravels, gravel-sand mixtures, little or no fines.
		Predominantly one size or a range of sizes with some intermediate sizes missing.	GP	Poorly graded gravels, gravel-sand mixtures, little or no fines.
	GRAVELS W/FINES (High % Fines)	Non-plastic fines (for identification procedures, see ML)	GM	Silty gravels, poorly graded gravel-sand-silt mixtures.
		Plastic fines (for identification procedures, see CL)	GC	Clayey gravels, poorly graded gravel-sand-clay mixtures.
SANDS 50% or more of coarse fraction < No. 4 sieve	CLEAN SANDS (Low % Fines)	Wide range in grain size and substantial amounts of all intermediate particle sizes.	SW	Well graded sand, gravelly sands, little or no fines.
		Predominantly one size or a range of sizes with some intermediate sizes missing.	SP	Poorly graded sands, gravelly sands, little or no fines.
	SANDS W/FINES (High % Fines)	Non-plastic fines (for identification procedures, see ML)	SM	Silty sands, poorly graded sand-silt mixtures.
		Plastic fines (for identification procedures, see CL)	SC	Clayey sands, poorly graded sand-clay mixtures.

FINE-GRAINED SOILS
More Than Half of Material is SMALLER Than No. 200 Sieve Size

FIELD IDENTIFICATION PROCEDURES (Excluding Particles Larger Than 3 Inches and Basing Fractions on Estimated Weights) – Identification Procedures on Fraction Smaller than No. 40 Sieve Size	DRY STRENGTH (Crushing Characteristics)	DILATANCY (Reaction to Shaking)	TOUGHNESS (Consistency Near Plastic Limit)	GROUP SYMBOL	TYPICAL NAMES
SILTS AND CLAYS Liquid Limit <50	None to Slight	Quick to Slow	None	ML	Inorganic silts and very fine sands, rock flour, silty or clayey fine sands with slight plasticity.
	Medium to High	None to Very Slow	Medium	CL	Inorganic clays of low to medium plasticity, gravelly clays, sandy clays, silty clays, lean clays.
	Slight to Medium	Slow	Slight	OL	Organic silts and organic silt-clays of low plasticity.
SILTS AND CLAYS Liquid Limit >50	Slight to Medium	Slow to None	Slight to Medium	MH	Inorganic silts, micaceous or diatomaceous fine sandy or silty soils, elastic silts.
	High to Very High	None	High	CH	Inorganic clays of high plasticity, fat clays.
	Medium to High	None to Very Slow	Slight to Medium	OH	Organic clays of medium to high plasticity.
HIGHLY ORGANIC SOILS	Readily identified by color, odor, spongy feel and frequently by fibrous texture.			Pt	Peat and other organic soils

Boundary classifications: Soils possessing characteristics of two groups are designated by combining group symbols. For example, GW-GC, well graded gravel-sand mixture with clay binder.
All sieve sizes on this chart are U.S. Standard.

DENSITY OF GRANULAR SOILS

DESIGNATION	STANDARD PENETRATION RESISTANCE-BLOWS/FOOT
Very Loose	0-4
Loose	5-10
Medium Loose	11-30
Dense	31-50
Very Dense	Over 50

CONSISTENCY OF COHESIVE SOILS

CONSISTENCY	UNC. COMPRESSIVE STRENGTH (TONS/SQ. FT.)	STANDARD PENETRATION RESISTANCE-BLOWS/FOOT	FIELD IDENTIFICATION METHODS
Very Soft	Less than 0.25	0 to 2	Easily penetrated several inches by fist
Soft	0.25 to 0.50	2 to 4	Easily penetrated several inches by thumb.
Medium Stiff	0.50 to 1.0	4 to 8	Can be penetrated several inches by thumb.
Stiff	1.0 to 2.0	8 to 15	Readily indented by thumb.
Very Stiff	2.0 to 4.0	15 to 30	Readily indented by thumbnail.
Hard	More than 4.0	Over 30	Indented with difficulty by thumbnail.

ROCK TERMS

ROCK HARDNESS (FROM CORE SAMPLES)			ROCK BROKENNESS		
Descriptive Terms	Screwdriver or Knife Effects	Hammer Effects	Descriptive Terms	Abbreviation	Spacing
Soft	Easily Gouged	Crushes when pressed with hammer	Very Broken	(V. Br.)	0-2"
Medium Soft	Can be Gouged	Breaks (one blow); crumbly edges	Broken	(Br.)	2"-1'
Medium Hard	Can be scratched	Breaks (one blow); sharp edges	Blocky	(Bl.)	1'-3'
Hard	Cannot be scratched	Breaks conchoidally (several blows); sharp edges	Massive	(M.)	3'-10'

LEGEND:

SOIL SAMPLES - TYPES

S - 2" Split-Barrel Sample
ST - 3" O.D. Undisturbed Sample
O - Other Samples, Specify in Remarks

ROCK SAMPLES - TYPES

X - NX (Conventional) Core (~2-1/8" O.D.)
Q - NQ (Wireline) Core (~1-7/8" O.D.)
Z - Other Core Sizes, Specify in Remarks

WATER LEVELS

12/18 ▼ 12.6' Initial Level w/Date & Depth
12/18 ▼ 12.6' Stabilized Level w/Date & Depth

5.2.1 USCS Classification

Soils are to be classified according to the Unified Soil Classification System (USCS). This method of classification is detailed in Figure 1 (Continued).
This method of classification identifies soil types on the basis of grain size and cohesiveness.

Fine-grained soils, or fines, are smaller than the No. 200 sieve and are of two types: silt (M) and clay (C). Some classification systems define size ranges for these soil particles, but for field classification purposes, they are identified by their respective behaviors. Organic material (O) is a common component of soil but has no size range; it is recognized by its composition. The careful study of the USCS will aid in developing the competence and consistency necessary for the classification of soils.

Coarse-grained soils shall be divided into rock fragments, sand, or gravel. The terms sand and gravel not only refer to the size of the soil particles but also to their depositional history. To insure accuracy in description, the term rock fragments shall be used to indicate angular granular materials resulting from the breakup of rock. The sharp edges typically observed indicate little or no transport from their source area, and therefore the term provides additional information in reconstructing the depositional environment of the soils encountered. When the term "rock fragments" is used it shall be followed by a size designation such as "(1/4 inchΦ-1/2 inchΦ)" or "coarse-sand size" either immediately after the entry or in the remarks column. The USCS classification would not be affected by this variation in terms.

5.2.2 Color

Soil colors shall be described utilizing a single color descriptor preceded, when necessary, by a modifier to denote variations in shade or color mixtures. A soil could therefore be referred to as "gray" or "light gray" or "blue-gray." Since color can be utilized in correlating units between sampling locations, it is important for color descriptions to be consistent from one boring to another.

Colors must be described while the sample is still moist. Soil samples shall be broken or split vertically to describe colors. Samplers tend to smear the sample surface creating color variations between the sample interior and exterior.

The term "mottled" shall be used to indicate soils irregularly marked with spots of different colors. Mottling in soils usually indicates poor aeration and lack of good drainage.

Soil Color Charts shall not be used unless specified by the project manager.

5.2.3 Relative Density and Consistency

To classify the relative density and/or consistency of a soil, the geologist is to first identify the soil type. Granular soils contain predominantly sands and gravels. They are noncohesive (particles do not adhere well when compressed). Finer-grained soils (silts and clays) are cohesive (particles will adhere together when compressed).

The density of noncohesive, granular soils is classified according to standard penetration resistances obtained from split-barrel sampling performed according to the methods detailed in Standard Operating Procedures GH-1.3 and SA-1.3. Those designations are:

Designation	Standard Penetration Resistance (Blows per Foot)
Very loose	0 to 4
Loose	5 to 10
Medium dense	11 to 30
Dense	31 to 50
Very dense	Over 50

Standard penetration resistance is the number of blows required to drive a split-barrel sampler with a 2-inch outside diameter 12 inches into the material using a 140-pound hammer falling freely through 30 inches. The sampler is driven through an 18-inch sample interval, and the number of blows is recorded for each 6-inch increment. The density designation of granular soils is obtained by adding the number of blows required to penetrate the last 12 inches of each sample interval. It is important to note that if gravel or rock fragments are broken by the sampler or if rock fragments are lodged in the tip, the resulting blow count will be erroneously high, reflecting a higher density than actually exists. This shall be noted on the log and referenced to the sample number. Granular soils are given the USCS classifications GW, GP, GM, SW, SP, SM, GC, or SC (see Figure 1).

The consistency of cohesive soils is determined by performing field tests and identifying the consistency as shown in Figure 2.

Cohesive soils are given the USCS classifications ML, MH, CL, CH, OL, or OH (see Figure 1).

The consistency of cohesive soils is determined either by blow counts, a pocket penetrometer (values listed in the table as Unconfined Compressive Strength), or by hand by determining the resistance to penetration by the thumb. The pocket penetrometer and thumb determination methods are conducted on a selected sample of the soil, preferably the lowest 0.5 foot of the sample in the split-barrel sampler. The sample shall be broken in half and the thumb or penetrometer pushed into the end of the sample to determine the consistency. Do not determine consistency by attempting to penetrate a rock fragment. If the sample is decomposed rock, it is classified as a soft decomposed rock rather than a hard soil. Consistency shall not be determined solely by blow counts. One of the other methods shall be used in conjunction with it. The designations used to describe the consistency of cohesive soils are shown in Figure 2.

5.2.4 Weight Percentages

In nature, soils are comprised of particles of varying size and shape, and are combinations of the various grain types. The following terms are useful in the description of soil:

Terms of Identifying Proportion of the Component	Defining Range of Percentages by Weight
Trace	0 - 10 percent
Some	11 - 30 percent
Adjective form of the soil type (e.g., "sandy")	31 - 50 percent

FIGURE 2

CONSISTENCY FOR COHESIVE SOILS

Consistency	Standard Penetration Resistance (Blows per Foot)	Unconfined Compressive Strength (Tons/Sq. Foot by pocket penetration)	Field Identification
Very soft	0 to 2	Less than 0.25	Easily penetrated several inches by fist
Soft	2 to 4	0.25 to 0.50	Easily penetrated several inches by thumb
Medium stiff	4 to 8	0.50 to 1.0	Can be penetrated several inches by thumb with moderate effort
Stiff	8 to 15	1.0 to 2.0	Readily indented by thumb but penetrated only with great effort
Very stiff	15 to 30	2.0 to 4.0	Readily indented by thumbnail
Hard	Over 30	More than 4.0	Indented with difficulty by thumbnail

Examples:

- Silty fine sand: 50 to 69 percent fine sand, 31 to 50 percent silt.
- Medium to coarse sand, some silt: 70 to 80 percent medium to coarse sand, 11 to 30 percent silt.
- Fine sandy silt, trace clay: 50 to 68 percent silt, 31 to 49 percent fine sand, 1 to 10 percent clay.
- Clayey silt, some coarse sand: 70 to 89 percent clayey silt, 11 to 30 percent coarse sand.

5.2.5 Moisture

Moisture content is estimated in the field according to four categories: dry, moist, wet, and saturated. In dry soil, there appears to be little or no water. Saturated samples obviously have all the water they can hold. Moist and wet classifications are somewhat subjective and often are determined by the individual's judgment. A suggested parameter for this would be calling a soil wet if rolling it in the hand or on a porous surface liberates water, i.e., dirties or muddies the surface. Whatever method is adopted for describing moisture, it is important that the method used by an individual remains consistent throughout an entire drilling job.

Laboratory tests for water content shall be performed if the natural water content is important.

5.2.6 Stratification

Stratification can only be determined after the sample barrel is opened. The stratification or bedding thickness for soil and rock is depending on grain size and composition. The classification to be used for stratification description is shown in Figure 3.

5.2.7 Texture/Fabric/Bedding

The texture/fabric/bedding of the soil shall be described. Texture is described as the relative angularity of the particles: rounded, subrounded, subangular, and angular. Fabric shall be noted as to whether the particles are flat or bulky and whether there is a particular relation between particles (i.e., all the flat particles are parallel or there is some cementation). The bedding or structure shall also be noted (e.g., stratified, lensed, nonstratified, heterogeneous varved).

5.2.8 Summary of Soil Classification

In summary, soils shall be classified in a similar manner by each geologist/engineer at a project site. The hierarchy of classification is as follows:

- Density and/or consistency
- Color
- Plasticity (Optional)
- Soil types
- Moisture content
- Stratification
- Texture, fabric, bedding
- Other distinguishing features

FIGURE 3

BEDDING THICKNESS CLASSIFICATION

Thickness (metric)	Thickness (Approximate English Equivalent)	Classification
> 1.0 meter	> 3.3'	Massive
30 cm - 1 meter	1.0' - 3.3'	Thick Bedded
10 cm - 30 cm	4" - 1.0'	Medium Bedded
3 cm - 10 cm	1" - 4"	Thin Bedded
1 cm - 3 cm	2/5" - 1"	Very Thin Bedded
3 mm - 1 cm	1/8" - 2/5"	Laminated
1 mm - 3 mm	1/32" - 1/8"	Thinly Laminated
< 1 mm	<1/32"	Micro Laminated

(Weir, 1973 and Ingram, 1954)

5.3 Classification of Rocks

Rocks are grouped into three main divisions: sedimentary, igneous and metamorphic. Sedimentary rocks are by far the predominant type exposed at the earth's surface. The following basic names are applied to the types of rocks found in sedimentary sequences:

- Sandstone - Made up predominantly of granular materials ranging between 1/16 to 2 mm in diameter.
- Siltstone - Made up of granular materials less than 1/16 to 1/256 mm in diameter. Fractures irregularly. Medium thick to thick bedded.
- Claystone - Very fine-grained rock made up of clay and silt-size materials. Fractures irregularly. Very smooth to touch. Generally has irregularly spaced pitting on surface of drilled cores.
- Shale - A fissile very fine-grained rock. Fractures along bedding planes.
- Limestone - Rock made up predominantly of calcite ($CaCO_3$). Effervesces strongly upon the application of dilute hydrochloric acid.
- Coal - Rock consisting mainly of organic remains.
- Others - Numerous other sedimentary rock types are present in lesser amounts in the stratigraphic record. The local abundance of any of these rock types is dependent upon the depositional history of the area. Conglomerate, halite, gypsum, dolomite, anhydrite, lignite, etc. are some of the rock types found in lesser amounts.

In classifying a sedimentary rock the following hierarchy shall be noted:

- Rock type
- Color
- Bedding thickness
- Hardness
- Fracturing
- Weathering
- Other characteristics

5.3.1 Rock Type

As described above, there are numerous types of sedimentary rocks. In most cases, a rock will be a combination of several grain types, therefore, a modifier such as a sandy siltstone, or a silty sandstone can be used. The modifier indicates that a significant portion of the rock type is composed of the modifier. Other modifiers can include carbonaceous, calcareous, siliceous, etc.

Grain size is the basis for the classification of clastic sedimentary rocks. Figure 4 is the Udden-Wentworth classification that will be assigned to sedimentary rocks. The individual boundaries are slightly different than the USCS subdivision for soil classification. For field determination of grain sizes, a scale can be used for the coarse grained rocks. For example, the division between siltstone and claystone may not be measurable in the field. The boundary shall be determined by use of a hand lens. If the grains cannot be seen with the naked eye but are distinguishable with a hand lens, the rock is a siltstone. If the grains are not distinguishable with a hand lens, the rock is a claystone.

FIGURE 4

GRAIN SIZE CLASSIFICATION FOR ROCKS

Particle Name	Grain Size Diameter
Cobbles	> 64 mm
Pebbles	4 - 64 mm
Granules	2 - 4 mm
Very Coarse Sand	1 - 2 mm
Coarse Sand	0.5 - 1 mm
Medium Sand	0.25 - 0.5 mm
Fine Sand	0.125 - 0.25 mm
Very Fine Sand	0.0625 - 0.125 mm
Silt	0.0039 - 0.0625 mm

After Wentworth, 1922

5.3.2 Color

The color of a rock can be determined in a similar manner as for soil samples. Rock core samples shall be classified while wet, when possible, and air cored samples shall be scraped clean of cuttings prior to color classifications.

Rock color charts shall not be used unless specified by the Project Manager.

5.3.3 Bedding Thickness

The bedding thickness designations applied to soil classification (see Figure 3) will also be used for rock classification.

5.3.4 Hardness

The hardness of a rock is a function of the compaction, cementation, and mineralogical composition of the rock. A relative scale for sedimentary rock hardness is as follows:

- Soft - Weathered, considerable erosion of core, easily gouged by screwdriver, scratched by fingernail. Soft rock crushes or deforms under pressure of a pressed hammer. This term is always used for the hardness of the saprolite (decomposed rock which occupies the zone between the lowest soil horizon and firm bedrock).

- Medium soft - Slight erosion of core, slightly gouged by screwdriver, or breaks with crumbly edges from single hammer blow.

- Medium hard - No core erosion, easily scratched by screwdriver, or breaks with sharp edges from single hammer blow.

- Hard - Requires several hammer blows to break and has sharp conchoidal breaks. Cannot be scratched with screwdriver.

Note the difference in usage here of the works "scratch" and "gouge." A scratch shall be considered a slight depression in the rock (do not mistake the scraping off of rock flour from drilling with a scratch in the rock itself), while a gouge is much deeper.

5.3.5 Fracturing

The degree of fracturing or brokenness of a rock is described by measuring the fractures or joint spacing. After eliminating drilling breaks, the average spacing is calculated and the fracturing is described by the following terms:

- Very broken (V. BR.) - Less than 2-inch spacing between fractures
- Broken (BR.) - 2-inch to 1-foot spacing between fractures
- Blocky (BL.) - 1- to 3-foot spacing between fractures
- Massive (M.) - 3 to 10-foot spacing between fractures

The structural integrity of the rock can be approximated by calculating the Rock Quality Designation (RQD) of cores recovered. The RQD is determined by adding the total lengths of all pieces exceeding 4 inches and dividing by the total length of the coring run, to obtain a percentage.

Method of Calculating RQD
(After Deere, 1964)

$$RQD\ \% = r/l \times 100$$

r = Total length of all pieces of the lithologic unit being measured, which are greater than 4 inches length, and have resulted from natural breaks. Natural breaks include slickensides, joints, compaction slicks, bedding plane partings (not caused by drilling), friable zones, etc.

l = Total length of the coring run.

5.3.6 Weathering

The degree of weathering is a significant parameter that is important in determining weathering profiles and is also useful in engineering designs. The following terms can be applied to distinguish the degree of weathering:

- Fresh - Rock shows little or no weathering effect. Fractures or joints have little or no staining and rock has a bright appearance.
- Slight - Rock has some staining which may penetrate several centimeters into the rock. Clay filling of joints may occur. Feldspar grains may show some alteration.
- Moderate - Most of the rock, with exception of quartz grains, is stained. Rock is weakened due to weathering and can be easily broken with hammer.
- Severe - All rock including quartz grains is stained. Some of the rock is weathered to the extent of becoming a soil. Rock is very weak.

5.3.7 Other Characteristics

The following items shall be included in the rock description:

- Description of contact between two rock units. These can be sharp or gradational.
- Stratification (parallel, cross stratified).
- Description of any filled cavities or vugs.
- Cementation (calcareous, siliceous, hematitic).
- Description of any joints or open fractures.
- Observation of the presence of fossils.
- Notation of joints with depth, approximate angle to horizontal, any mineral filling or coating, and degree of weathering.

All information shown on the boring logs shall be neat to the point where it can be reproduced on a copy machine for report presentation. The data shall be kept current to provide control of the drilling program and to indicate various areas requiring special consideration and sampling.

5.3.8 Additional Terms Used in the Description of Rock

The following terms are used to further identify rocks:

- Seam - Thin (12 inches or less), probably continuous layer.
- Some - Indicates significant (15 to 40 percent) amounts of the accessory material. For example, rock composed of seams of sandstone (70 percent) and shale (30 percent) would be "sandstone -- some shale seams."
- Few - Indicates insignificant (0 to 15 percent) amounts of the accessory material. For example, rock composed of seam of sandstone (90 percent) and shale (10 percent) would be "sandstone -- few shale seams."
- Interbedded - Used to indicate thin or very thin alternating seams of material occurring in approximately equal amounts. For example, rock composed of thin alternating seams of sandstone (50 percent) and shale (50 percent) would be "interbedded sandstone and shale."
- Interlayered - Used to indicate thick alternating seams of material occurring in approximately equal amounts.

The preceding sections describe the classification of sedimentary rocks. The following are some basic names that are applied to igneous rocks:

- Basalt - A fine-grained extrusive rock composed primarily of calcic plagioclase and pyroxene.
- Rhyolite - A fine-grained volcanic rock containing abundant quartz and orthoclase. The fine-grained equivalent of a granite.
- Granite - A coarse-grained plutonic rock consisting essentially of alkali feldspar and quartz.
- Diorite - A coarse-grained plutonic rock consisting essentially of sodic plagioclase and hornblende.
- Gabbro - A coarse-grained plutonic rock consisting of calcic plagioclase and clinopyroxene. Loosely used for any coarse-grained dark igneous rock.

The following are some basic names that are applied to metamorphic rocks:

- Slate - A very fine-grained foliated rock possessing a well developed slaty cleavage. Contains predominantly chlorite, mica, quartz, and sericite.
- Phyllite - A fine-grained foliated rock that splits into thin flaky sheets with a silky sheen on cleavage surface.
- Schist - A medium to coarse-grained foliated rock with subparallel arrangement of the micaceous minerals which dominate its composition.
- Gneiss - A coarse-grained foliated rock with bands rich in granular and platy minerals.
- Quartzite - A fine- to coarse-grained nonfoliated rock breaking across grains, consisting essentially of quartz sand with silica cement.

5.4 Abbreviations

Abbreviations may be used in the description of a rock or soil. However, they shall be kept at a minimum. Following are some of the abbreviations that may be used:

C	-	Coarse	Lt	-	Light	Yl	-	Yellow
Med	-	Medium	BR	-	Broken	Or	-	Orange
F	-	Fine	BL	-	Blocky	SS	-	Sandstone
V	-	Very	M	-	Massive	Sh	-	Shale
Sl	-	Slight	Br	-	Brown	LS	-	Limestone
Occ	-	Occasional	Bl	-	Black	Fgr	-	Fine-grained
Tr	-	Trace						

5.5 Boring Logs and Documentation

This section describes in more detail the procedures to be used in completing boring logs in the field. Information obtained from the preceding sections shall be used to complete the logs. A sample boring log has been provided as Figure 5.

The field geologist/engineer shall use this example as a guide in completing each boring log. Each boring log shall be fully described by the geologist/engineer as the boring is being drilled. Every sheet contains space for 25 feet of log. Information regarding classification details is provided either on the back of the boring log or on a separate sheet, for field use.

5.5.1 Soil Classification

- Identify site name, boring number, job number, etc. Elevations and water level data to be entered when surveyed data is available.

- Enter sample number (from SPT) under appropriate column. Enter depth sample was taken from (1 block = 1 foot). Fractional footages, i.e., change of lithology at 13.7 feet, shall be lined off at the proportional location between the 13- and 14-foot marks. Enter blow counts (Standard Penetration Resistance) diagonally (as shown). Standard penetration resistance is covered in Section 5.2.3.

- Determine sample recovery/sample length as shown. Measure the total length of sample recovered from the split-spoon sampler, including material in the drive shoe. Do not include cuttings or wash material that may be in the upper portion of the sample tube.

- Indicate any change in lithology by drawing a line at the appropriate depth. For example, if clayey silt was encountered from 0 to 5.5 feet and shale from 5.5 to 6.0 feet, a line shall be drawn at this increment. This information is helpful in the construction of cross-sections. As an alternative, symbols may be used to identify each change in lithology.

- The density of granular soils is obtained by adding the number of blows for the last two increments. Refer to Density of Granular Soils Chart on back of log sheet. For consistency of cohesive soils refer also to the back of log sheet - Consistency of Cohesive Soils. Enter this information under the appropriate column. Refer to Section 5.2.3.

FIGURE 5
COMPLETED BORING LOG (EXAMPLE)

BOREHOLE LOG

Page 1 of 1

PROJECT NAME: NSB - SITE
PROJECT NUMBER: 9594
DRILLING COMPANY: SOILTEST CO.
DRILLING RIG: CME-55
BORING NUMBER: SB/MW 1
DATE: 3/8/96
GEOLOGIST: SJ CONTI
DRILLER: R. ROCK

Sample No. and Type or RQD	Depth (Ft.) or Run No.	Blows / 6" or RQD (%)	Sample Recovery / Sample Length	Lithology Change (Depth/Ft.) or Screened Interval	MATERIAL DESCRIPTION: Soil Density/ Consistency or Rock Hardness	Color	Material Classification	USCS *	Remarks	PID/FID Reading (ppm): Sample	Sampler BZ	Borehole**	Driller BZ**
S-1	0.0	7/6	1.5/2.0		M DENSE	BRN	SILTY SAND - SOME	SM	MOIST SL. ORG. ODOR	5	0	0	0
C 0800	2.0	9/10				TO BLK	ROCK FR - TR BRICKS		FILL TO 4'±				
							(FILL)						
	4.0			4.0									
S-2 C		5/7	2.9/2.0		M DENSE	BRN	SILTY SAND - TR FINE	SM	MOIST - W ODOR	10	10	-	-
0810	6.0	9/8					GRAVEL		NAT. MATL. TOOK SAMPLE				
	•			7' ▽					SB01-0406 FOR ANALYSIS				
	8.0			8.0									
S-3 C		6/8	1.9/2.0		DENSE	TAN BRN	FINE TO COARSE SAND	SW	WET	0	0	0	0
0820	10.0	17/16					TR. F. GRAVEL		HIT WATER @ 7'±				
	12.0			12.0									
S-4 C		7/6	1.6/2.0		STIFF	GRAY	SILTY CLAY	CL	MOIST → WET	0	-5	-	-
0830	14.0	5/8											
	15.0			15.0					AUGER REF @ 15'				
				LITHIC	M HARD	BRN	SILTSTONE	VBR	WEATHERED				
				16					LO ⅋ JNTS @ 15.5				
9/5	①		4.0/5.0						WATER STAINS @ 16.5, 17.1, 17.5	0	0	0	0
				19'					LOSING SOME				
	20.0				HARD	GRAY	SANDSTONE - SOME	BR	DRILL H2O @ 17'±				
							SILTSTONE		SET TEMP 6" CAS TO 15.5				
4.9/5.0	②		5.0/5.0						SET 2" Ø PVC SCREEN 16-25	0	0	0	0
									SAND 14-25				
	25.0			25					PELLETS 12-14				

* When rock coring, enter rock brokeness.

** Include monitor reading in 6 foot intervals @ borehole. Increase reading frequency if elevated response read.

• 1-20Z
1-80Z

Drilling Area Background (ppm): 0

Remarks: CME-55 R16, 4¼" ID HSA - 9" OD±
2" SPLIT SPOONS - 140 LB HAMMER - 30" DROP
NX CORE IN BEDROCK RUN ① = 25 min. RUN ② = 15 min

Converted to Well: Yes ✓ No ____ Well I.D. #: MW-1

- Enter color of the material in the appropriate column.
- Describe material using the USCS. Limit this column for sample description only. The predominant material is described last. If the primary soil is silt but has fines (clay) - use clayey silt. Limit soil descriptors to the following:
  - Trace: 0 - 10 percent
  - Some: 11 - 30 percent
  - And/Or: 31 - 50 percent
- Also indicate under Material Classification if the material is fill or natural soils. Indicate roots, organic material, etc.
- Enter USCS symbol - use chart on back of boring log as a guide. If the soils fall into one of two basic groups, a borderline symbol may be used with the two symbols separated by a slash. For example ML/CL or SM/SP.
- The following information shall be entered under the "Remarks" column and shall include, but is not limited by, the following:
  - Moisture - estimate moisture content using the following terms - dry, moist, wet and saturated. These terms are determined by the individual. Whatever method is used to determine moisture, be consistent throughout the log.
  - Angularity - describe angularity of coarse grained particles using the terms angular, subangular, subrounded, or rounded. Refer to ASTM D 2488 or Earth Manual for criteria for these terms.
  - Particle shape - flat, elongated, or flat and elongated.
  - Maximum particle size or dimension.
  - Water level observations.
  - Reaction with HCl - none, weak, or strong.
- Additional comments:
  - Indicate presence of mica, caving of hole, when water was encountered, difficulty in drilling, loss or gain of water.
  - Indicate odor and Photoionization Detector (PID) or Flame Ionization Detector (FID) reading if applicable.
  - Indicate any change in lithology by drawing a line through the lithology change column and indicate the depth. This will help when cross-sections are subsequently constructed.
  - At the bottom of the page indicate type of rig, drilling method, hammer size and drop, and any other useful information (i.e., borehole size, casing set, changes in drilling method).

- Vertical lines shall be drawn (as shown in Figure 5) in columns 6 to 8 from the bottom of each sample to the top of the next sample to indicate consistency of material from sample to sample, if the material is consistent. Horizontal lines shall be drawn if there is a change in lithology, then vertical lines drawn to that point.

- Indicate screened interval of well, as needed, in the lithology column. Show top and bottom of screen. Other details of well construction are provided on the well construction forms.

5.5.2 Rock Classification

- Indicate depth at which coring began by drawing a line at the appropriate depth. Indicate core run depths by drawing coring run lines (as shown) under the first and fourth columns on the log sheet. Indicate RQD, core run number, RQD percent, and core recovery under the appropriate columns.

- Indicate lithology change by drawing a line at the appropriate depth as explained in Section 5.5.1.

- Rock hardness is entered under designated column using terms as described on the back of the log or as explained earlier in this section.

- Enter color as determined while the core sample is wet; if the sample is cored by air, the core shall be scraped clean prior to describing color.

- Enter rock type based on sedimentary, igneous or metamorphic. For sedimentary rocks use terms as described in Section 5.3. Again, be consistent in classification. Use modifiers and additional terms as needed. For igneous and metamorphic rock types use terms as described in Sections 5.3.8.

- Enter brokenness of rock or degree of fracturing under the appropriate column using symbols VBR, BR, BL, or M as explained in Section 5.3.5 and as noted on the back of the Boring Log.

- The following information shall be entered under the remarks column. Items shall include but are not limited to the following:

  - Indicate depths of joints, fractures and breaks and also approximate to horizontal angle (such as high, low), i.e., 70° angle from horizontal, high angle.
  - Indicate calcareous zones, description of any cavities or vugs.
  - Indicate any loss or gain of drill water.
  - Indicate drop of drill tools or change in color of drill water.

- Remarks at the bottom of Boring Log shall include:

  - Type and size of core obtained.
  - Depth casing was set.
  - Type of rig used.

- As a final check the boring log shall include the following:

  - Vertical lines shall be drawn as explained for soil classification to indicate consistency of bedrock material.

  - If applicable, indicate screened interval in the lithology column. Show top and bottom of screen. Other details of well construction are provided on the well construction forms.

5.5.3 Classification of Soil and Rock from Drill Cuttings

The previous sections describe procedures for classifying soil and rock samples when cores are obtained. However, some drilling methods (air/mud rotary) may require classification and borehole logging based on identifying drill cuttings removed from the borehole. Such cuttings provide only general information on subsurface lithology. Some procedures that shall be followed when logging cuttings are:

- Obtain cutting samples at approximately 5-foot intervals, sieve the cuttings (if mud rotary drilling) to obtain a cleaner sample, place the sample into a small sample bottle or "zip lock" bag for future reference, and label the jar or bag (i.e. hole number, depth, date, etc.). Cuttings shall be closely examined to determine general lithology.
- Note any change in color of drilling fluid or cuttings, to estimate changes in lithology.
- Note drop or chattering of drilling tools or a change in the rate of drilling, to determine fracture locations or lithologic changes.
- Observe loss or gain of drilling fluids or air (if air rotary methods are used), to identify potential fracture zones.
- Record this and any other useful information onto the boring log as provided in Figure 1.

This logging provides a general description of subsurface lithology and adequate information can be obtained through careful observation of the drilling process. It is recommended that split-barrel and rock core sampling methods be used at selected boring locations during the field investigation to provide detailed information to supplement the less detailed data generated through borings drilled using air/mud rotary methods.

5.6 Review

Upon completion of the borings logs, copies shall be made and reviewed. Items to be reviewed include:

- Checking for consistency of all logs.
- Checking for conformance to the guideline.
- Checking to see that all information is entered in their respective columns and spaces.

6.0 REFERENCES

Unified Soil Classification System (USCS).

ASTM D2488, 1985.

Earth Manual, U.S. Department of the Interior, 1974.

7.0 RECORDS

Originals of the boring logs shall be retained in the project files.

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		Applicability Tetra Tech NUS, Inc.	
		Prepared Earth Sciences Department	
Subject SAMPLE NOMENCLATURE		Approved Tom Johnston	

TABLE OF CONTENTS

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to specify a consistent sample nomenclature system that will facilitate subsequent data management in a cost-effective manner. The sample nomenclature system has been devised such that the following objectives can be attained:

- Sorting of data by matrix
- Sorting of data by depth
- Maintenance of consistency (field, laboratory, and database sample numbers)
- Accommodation of all project-specific requirements
- Accommodation of laboratory sample number length constraints (maximum of 20 characters)

2.0 SCOPE

The methods described in this SOP shall be used consistently for all projects requiring electronic data. Other contract- or project-specific sample nomenclature requirements may also be applicable.

3.0 GLOSSARY

None.

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Program Manager - It shall be the responsibility of the Project Manager (or designee) to inform contract-specific Project Managers (PMs) of the existence and requirements of this SOP.

Project Manager - It shall be the responsibility of the PM to determine the applicability of this SOP based on: (1) program-specific requirements and (2) project size and objectives. It shall be the responsibility of the PM (or designee) to ensure that sample nomenclature requirements are thoroughly specified in the relevant project planning document (e.g., sampling and analysis plan) and are consistent with this SOP if relevant. It shall be the responsibility of the PM to ensure that the FOL is familiar with the sample nomenclature system.

Field Operations Leader (FOL) - It shall be the responsibility of the FOL to ensure that all field technicians or sampling personnel are thoroughly familiar with this SOP and the project-specific sample nomenclature system. It shall be the responsibility of the FOL to ensure that the sample nomenclature system is used during all project-specific sampling efforts.

General personnel qualifications for sample nomenclature activities in the field include the following:

- Occupational Safety and Health Administration (OSHA) 40-hour and applicable refresher training.
- Capability of performing field work under the expected physical and environmental (i.e., weather) conditions.
- Familiarity with appropriate procedures for field documentation, handling, packaging, and shipping.

5.0 PROCEDURES

5.1 INTRODUCTION

The sample identification (ID) system can consist of as few as eight but not more than 20 distinct alpha-numeric characters. The sample ID will be provided to the laboratory on the sample labels and chain-of-custody forms. The basic sample ID provided to the laboratory has three segments and shall be as follows, where "A" indicates "alpha," and "N" indicates "numeric":

A or N 3 or 4 Characters	**AAA 2 or 3 Characters**	**A or N 3 to 6 Characters**
Site Identifier	Sample Type	Sample Location

Additional segments may be added as needed. For example:

(1) Soil and sediment sample ID

A or N 3 or 4 Characters	**AAA 2 or 3 Characters**	**A or N 3 to 6 Characters**	**NNNN 4 Characters**
Site identifier	Sample type	Sample location	Sample depth

(2) Aqueous (groundwater or surface water) sample ID

A or N 3 or 4 Characters	**AAA 2 or 3 Characters**	**A or N 3 to 6 Characters**	**NN 2 Characters**	**-A 1 Character**
Site identifier	Sample type	Sample location	Round number	Filtered sample only

(3) Biota sample ID

A or N 3 or 4 Characters	**AAA 2 or 3 Characters**	**A or N 3 to 6 Characters**	**AA 2 Characters**	**NNN 3 Characters**
Site identifier	Sample type	Sample location	Species identifier	Sample group number

5.2 SAMPLE IDENTIFICATION FIELD REQUIREMENTS

The various fields in the sample ID include but are not limited to the following:

- Site identifier
- Sample type
- Sample location
- Sample depth
- Sampling round number
- Filtered
- Species identifier
- Sample group number

The site identifier must be a three- or four-character field (numeric characters, alpha characters, or a mixture of alpha and numeric characters may be used). A site number is necessary because many facilities/sites have multiple individual sites, Solid Waste Management Units (SWMUs), Operable Units (OUs), etc. Several examples are presented in Section 5.3 of this SOP.

The sample type must be a two- or three-character alpha field. Suggested codes are provided in Section 5.3 of this SOP.

The sample location must be at least a three-character field but may have up to six characters (alpha, numeric, or a mixture). The six characters may be useful in identifying a monitoring well to be sampled or describing a grid location.

The sample depth field is used to note the depth below ground surface (bgs) at which a soil or sediment sample is collected. The first two numbers of the four-number code specify the top interval, and the third and fourth specify the bottom interval in feet bgs of the sample. If the sample depth is equal to or greater than 100, then only the top interval would be represented and the sampling depth would be truncated to three characters. The depths will be noted in whole numbers only; further detail, if needed, will be recorded on the sample log sheet or boring log, in the logbook, etc.

A two-digit round number will be used to track the number of aqueous samples collected from a particular aqueous sample location. The first sample collected from a location will be assigned the round identifier 01, the second 02, etc. This applies to both existing and proposed monitoring wells and surface water locations.

Aqueous samples that are field filtered (dissolved analysis) will be identified with an "-F" in the last field segment. No entry in this segment signifies an unfiltered (total) sample.

The species identifier must be a two-character alpha field. Several suggested codes are provided in Section 5.3 of this SOP.

The three-digit sample group number will be used to track the number of biota sample groups (a particular group size may be determined by sample technique, media type, the number of individual caught, weight issues, time, etc.) by species and location. The first sample group of a particular species collected from a given location will be assigned the sample group number 001, and the second sample group of the same species collected from the same location will be assigned the sample group number 002.

5.3 EXAMPLE SAMPLE FIELD DESIGNATIONS

Examples of each of the fields are as follows:

Site identifier - Examples of site numbers/designations are as follows:

A01 - Area of Concern (AOC) 1
125 - SWMU 125
000 - Base- or facility-wide sample (e.g., upgradient well)
BBG - Base background

The examples cited are only suggestions. Each PM (or designee) must designate appropriate (and consistent) site designations for their individual project.

Sample type - Examples of sample types are as follows:

AH - Ash Sample

AS - Air Sample
BM - Building Material Sample
BSB - Biota Sample Full Body
BSF - Biota Sample Fillet
CP - Composite Sample
CS - Chip Sample
DS - Drum Sample
DU - Dust Sample
FP - Free Product
IDW - Investigation-Derived Waste Sample
LT - Leachate Sample
MW - Monitoring Well Groundwater Sample
OF - Outfall Sample
RW - Residential Well Sample
SB - Soil Boring Sample
SD - Sediment Sample
SC - Scrape Sample
SG - Soil Gas Sample
SL - Sludge Sample
SP - Seep Sample
SS - Surface Soil Sample
ST - Storm Sewer Water Sample
SW - Surface Water Sample
TP - Test Pit Sample
TW - Temporary Well Sample
WC - Well Construction Material Sample
WP - Wipe Sample
WS - Waste/Solid Sample
WW - Wastewater Sample

Sample location - Examples of the location field are as follows:

001 - Monitoring well 1
N32E92 - Grid location 32 North and 92 East
D096 - Investigation-derived waste drum number 96

Species identifier - Examples of species identifier are as follows:

BC - Blue Crab
GB - Blue Gill
CO - Corn
SB - Soybean

5.4 EXAMPLES OF SAMPLE NOMENCLATURE

The first round monitoring well groundwater sample collected from existing monitoring well 001 at SWMU 16 for a filtered sample would be designated as 016MW00101-F.

The second round monitoring well groundwater sample collected from existing monitoring well C20P2 at Site 23 for an unfiltered sample would be designated as 023MWC20P202.

The second surface water sample collected from point 01 at SWMU 130 for an unfiltered sample would be designated as 130SW00102.

A surface soil sample collected from grid location 32 North and 92 East at Site 32 at the 0- to 2-foot interval would be designated as 032SSN32E920002.

A subsurface soil sample from soil boring 03 at SWMU 32 at an interval of 4 to 5 feet bgs would be designated as 032SB0030405.

A sediment sample collected at SWMU 19 from 0 to 6 inches at location 14 would be designated as 019SD0140001. The sample data sheet would reflect the precise depth at which this sample was collected.

During biota sampling for full-body analysis, the first time a minnow trap was checked at grid location A25 of SWMU 1415, three small blue gills were captured, collected, and designated with the sample ID of 1415BSBA25BG001. The second time blue gill were collected at the same location (grid location A25 at SWMU 1415), the sample ID would be 1415BSBA25BG002.

Note: No dash (-) or spacing is used between the segments with the exception of the filtered segment. The "F" used for a filtered aqueous sample is preceded by a dash (-F).

5.5 FIELD QA/QC SAMPLE NOMENCLATURE

Field Quality Assurance (QA)/Quality Control (QC) samples are designated using a different coding system. The QC code will consist of a three- to four-segment alpha-numeric code that identifies the sample QC type, the date the sample was collected, and the number of this type of QC sample collected on that date.

AA	NNNNNN	NN	-F
QC type	Date	Sequence number (per day)	Filtered (aqueous only, if needed)

The QC types are identified as:

TB = Trip Blank
RB = Rinsate Blank (Equipment Blank)
FD = Field Duplicate
AB = Ambient Conditions Blank
WB = Source Water Blank

The sampling time recorded on the chain-of-custody form, labels, and tags for duplicate samples will be 0000 so that the samples are "blind" to the laboratory. Notes detailing the sample number, time, date, and type will be recorded on the routine sample log sheets and will document the location of the duplicate sample (sample log sheets are not provided to the laboratory). Documentation for all other QC types (TB, RB, AB, and WB) will be recorded on the QC Sample Log Sheet (see SOP SA-6.3, Field Documentation).

5.6 EXAMPLES OF FIELD QA/QC SAMPLE NOMENCLATURE

The first duplicate of the day for a filtered groundwater sample collected on June 3, 2000, would be designated as FD06030001-F.

The third duplicate of the day taken of a subsurface soil sample collected on November 17, 2003, would be designated as FD11170303.

The first trip blank associated with samples collected on October 12, 2000, would be designated as TB10120001.

The only rinsate blank collected on November 17, 2001, would be designated as RB11170101.

6.0 DEVIATIONS

Any deviation from this SOP must be addressed in detail in the site-specific planning documents.

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		Effective Date 01/29/01	Revision 2
		Applicability Tetra Tech NUS, Inc.	
		Prepared Management Information Systems Department	
Subject DATABASE RECORDS AND QUALITY ASSURANCE		Approved D. Senovich	

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1.0 PURPOSE

The purpose of this document is to specify a consistent procedure for the quality assurance review of electronic and hard copy databases. This SOP outlines the requirements for establishment of a Database Record File, Quality Assurance review procedures, and documentation of the Quality Assurance Review Process.

2.0 SCOPE

The methods described in this Standard Operating Procedure (SOP) shall be used consistently for all projects managed by Tetra Tech NUS (TtNUS).

3.0 GLOSSARY

Chain-of-Custody Form - A Chain-of-Custody Form is a printed form that accompanies a sample or a group of samples from the time of sample collection to the laboratory. The Chain-of-Custody Form is retained with the samples during transfer of samples from one custodian to another. The Chain-of-Custody Form is a controlled document that becomes part of the permanent project file. Chain-of-Custody and field documentation requirements are addressed in SOP SA-6.1.

Electronic Database - A database provided on a compact laser disk (CD). Such electronic databases will generally be prepared using public domain software such as DBase, RBase, Oracle, Visual FoxPro, Microsoft Access, Paradox, etc.

Hardcopy Database - A printed copy of a database prepared using the software discussed under the definition of an electronic database.

Form I - A printed copy of the analytical results for each sample.

Sample Tracking Summary - A printed record of sample information including the date the samples were collected, the number of samples collected, the sample matrix, the laboratory to which the samples were shipped, the associated analytical requirements for the samples, the date the analytical data were received from the laboratory, and the date that validation of the sample data was completed.

4.0 RESPONSIBILITIES

Database Records Custodian - It shall be the responsibility of the Database Records Custodian to update and file the Sample Tracking Summaries for all active projects on a weekly basis. It shall be the responsibility of the Database Records Custodian to ensure that the most recent copies of the Sample Tracking Summaries are placed in the Database Records file. It shall be the responsibility of the Database Records Custodian to ensure that a copy of all validation deliverables is provided to the Project Manager (for placement in the project file). It shall be the responsibility of the Database Records Custodian to ensure that photocopies of all validation deliverables and historical data and reports (as applicable) are placed in the Database Records file.

Data Validation Coordinator - It shall be the responsibility of the Data Validation Coordinator (or designee) to ensure that the Sample Tracking Summaries are maintained by the Database Records Custodian. It shall be the responsibility of the Data Validation Coordinator (or designee) to ensure that photocopies of all data validation deliverables are placed in the applicable Database Records file by the Database Records Custodian.

Earth Sciences Department Manager - It shall be the responsibility of the Earth Sciences Department Manager (or equivalent) to ensure that all field personnel are familiar with the requirements of this Standard Operating Procedure (specifically Section 5.5).

FOL - It shall be the responsibility of the FOL (FOL) of each project to ensure that all field technicians or sampling personnel are thoroughly familiar with this SOP, specifically regarding provision of the Chain-of-Custody Forms to the Database Records Custodian. Other responsibilities of the FOL are described in Sections 5.4 and 5.5.

Management Information Systems (MIS) Manager - It shall be the responsibility of the MIS Manager to ensure that copies of original electronic deliverables (CDs) are placed in both the project files and the Database Records File. It shall be the responsibility of the MIS Manager (or designee) to verify the completeness of the database (presence of all samples) in both electronic and hardcopy form in the Database Records File. It shall be the responsibility of the MIS Manager to ensure that Quality Assurance Reviews are completed and are attested to by Quality Assurance Reviewers. It shall be the responsibility of the MIS Manager to ensure that records of the Quality Assurance review process are placed in the Database Records File. It shall be the responsibility of the MIS Manager to ensure that both electronic and hardcopy forms of the final database are placed in both the project and the Database Record File. It shall be the responsibility of the MIS Manager to ensure that data validation qualifiers are entered in the database.

Furthermore, it shall be the responsibility of the MIS Manager to participate in project planning at the request of the Project Manager, specifically with respect to the generation of level of effort and schedule estimates. To support the project planning effort, the MIS Manager shall provide a copy of the MIS Request From included as Attachment A to the project manager. It shall be the responsibility of the MIS Manager to generate level of effort and budget estimates at the time database support is requested if a budget does not exist at the time of the request. The MIS Request Form shall be provided to the Project Manager at the time of any such requests. It shall be the responsibility of the MIS Manager to notify the Project Manager of any anticipated level of effort overruns or schedule noncompliances as soon as such problems arise along with full justification for any deviations from the budget estimates (provided they were generated by the MIS Manager). It shall be the responsibility of the MIS Manager to document any changes to the scope of work dictated by the Project Manager, along with an estimate of the impact of the change on the level of effort and the schedule.

Program/Department Managers - It shall be the responsibility of the Department and/or Program Managers (or designees) to inform their respective department's Project Managers of the existence and requirements of this SOP.

Project Manager - It shall be the responsibility of each Project Manager to determine the applicability of this SOP based on: (1) program-specific requirements, and (2) project size and objectives. It shall be the responsibility of the Project Manager (or designee) to ensure that the FOL is familiar with the requirements regarding Chain-of-Custody Form provision to the Database Records Custodian. It shall be the responsibility of the Project Manager (or designee) to determine which, if any, historical data are relevant and to ensure that such data (including all relevant information such as originating entity, sample locations, sampling dates, etc.) are provided to the Database Records Custodian for inclusion in the Database Records File. It shall be the responsibility of the Project Manager to obtain project planning input regarding the level of effort and schedule from the MIS Manager. It shall be the responsibility of the Project Manager to complete the database checklist (Attachment A) to support the level of effort and schedule estimate and to facilitate database preparation and subroutine execution.

Risk Assessment Department Manager - It shall be the responsibility of the Risk Assessment Department Manager to monitor compliance with this Standard Operating Procedure, to modify this SOP as necessary, and to take corrective action if necessary. Monitoring of the process shall be completed on a quarterly basis.

Quality Assurance Reviewers - It shall be the responsibility of the Quality Assurance Reviewers to verify the completeness of the sample results via review of the Chain-of-Custody Forms and Sample Tracking Summaries. It shall be the responsibility of the Quality Assurance Reviewers to ensure the correctness of the database via direct comparison of the hardcopy printout of the database and the hardcopy summaries of the original analytical data (e.g., Form Is provided in data validation deliverables). Correctness includes the presence of all relevant sample information (all sample information fields), agreement of the laboratory and database analytical results, and the presence of data validation qualifiers.

Quality Manager - It shall be the responsibility of the Quality Manager to monitor compliance with this Standard Operating Procedure via routine audits.

5.0 PROCEDURES

5.1 Introduction

Verification of the accuracy and completeness of an electronic database can only be accomplished via comparison of a hardcopy of the database with hardcopy of all relevant sample information. The primary purposes of this SOP are to ensure that 1) all necessary hardcopy information is readily available to Quality Assurance Reviewers; 2) ensure that the Quality Assurance review is completed in a consistent and comprehensive manner, and; 3) ensure that documentation of the Quality Assurance review process is maintained in the project file.

5.2 File Establishment

A Database Record file shall be established for a specific project at the discretion of the Project Manager. Initiation of the filing procedure will commence upon receipt of the first set of Chain-of-Custody documents from a FOL or sampling technician. The Database Record Custodian shall establish a project-specific file for placement in the Database Record File. Each file in the Database Record File shall consist of standard components placed in the file as the project progresses. Each file shall be clearly labeled with the project number, which shall be placed on the front of the file drawer and on each and every hanging file folder relevant to the project. The following constitute the minimum components of a completed file:

- Electronic Deliverables
- Sample Tracking Forms
- Chain-of-Custody Forms
- Data Validation Letters
- Quality Assurance Records

5.3 Electronic Deliverables

The format of electronic deliverables shall be specified in the laboratory procurement specification and shall be provided by the laboratory. The integrity of all original electronic data deliverables shall be maintained. This shall be accomplished via the generation of copies of each electronic deliverable provided by the laboratory. The original electronic deliverable shall be provided to the project manager for inclusion in the project file. A copy of the original electronic deliverable shall be placed in the Database Record File. The second copy shall be maintained by the MIS Manager (or designee) to be used as a working copy.

5.4 Sample Tracking Forms

Updated versions of the sample tracking form for each relevant project shall be maintained by the Database Record Custodian. The Sample Tracking Forms shall be updated any time additional Chain-of-Custody Forms are received from a FOL or sampling technician, or at any time that data are received from a laboratory, or at any time that validation of a given data package (sample delivery group) is completed. The Data Validation Coordinator shall inform the Database Record Custodian of the receipt of any data packages from the laboratory and of completion of validation of a given data package to facilitate updating of the Sample Tracking Form. The Database Record Custodian shall place a revised copy of the Sample Tracking Form in the Database Record File anytime it has been updated. Copies of the updated Sample Tracking Form shall also be provided to the project manager to apprise the project manager of sample package receipt, completion of validation, etc.

5.5 Chain-of-Custody Forms

The Chain-of-Custody Forms for all sampling efforts will be used as the basis for (1) updating the Sample Tracking Form, and (2) confirming that all required samples and associated analyses have been completed. It shall be the responsibility of the FOL (or sample technician) to provide a photocopy of all Chain-of-Custody Forms to the Database Record Custodian immediately upon completion of a sampling effort. The Database Record Custodian shall then place the copies of the Chain-of-Custody Form(s) in the Database Record File. Upon receipt of a sample data package from an analytical laboratory, the Data Validation Coordinator shall provide a copy of the laboratory Chain-of-Custody Form to the Database Record Custodian. The Database Record Custodian shall use this copy to update the Sample Tracking Summary and shall place the copy of the laboratory-provided Chain-of-Custody Form in the Database Record File. The photocopy of the laboratory-provided Chain-of Custody Form shall be stapled to the previously filed field copy. Upon receipt of all analytical data, two copies of the Chain-of-Custody will therefore be in the file. Review of the Chain-of-Custody Forms will therefore be a simple mechanism to determine if all data have been received. Chain-of-Custody is addressed in SOP SA-6.1.

5.6 Data Validation Letters

All data validation deliverables (or raw data summaries if validation is not conducted) shall be provided for inclusion in both the Database Record File and the project file. If USEPA regional- or client-specific requirements are such that Form Is (or similar analytical results) need not be provided with the validation deliverable, copies of such results must be appended to the deliverable. It is preferable, although not essential that the validation qualifiers be hand-written directly on the data summary forms. The data validation deliverables (and attendant analytical summaries) will provide the basis for direct comparison of the database printout and the raw data and qualifiers.

5.7 Historical Data

At the direction of the Project Manager, historical data may also be included in a project-specific analytical database. In the event that historical data are germane to the project, hardcopy of the historical data must be included in the Database Record File. Historical data may be maintained in the form of final reports or as raw data. The information contained in the historical data file must be sufficient to identify its origin, its collection date, the sample location, the matrix, and any and all other pertinent information. All available analytical data, Chain-of-Custody Forms, boring logs, well construction logs, sample location maps, shall be photocopied by the Project Manager (or designee) and placed in one or more 3-ring binders. All information shall be organized chronologically by matrix. It shall be the responsibility of the Project Manager (or designee) to ensure that all inconsistencies between analytical data, Chain-of-Custody Forms, boring logs, sample log sheets, and field logbooks are identified and corrected. The Project Manager (or designee) shall decide which nomenclature is appropriate and edit, initial and date all relevant forms. Data entry may only be performed on information that has undergone the aforementioned

editing process, thereby having a direct correlation between hardcopy information and what will become the electronic database.

6.0 RECORDS

Records regarding database preparation and quality assurance review include all those identified in the previous section. Upon completion of the database task, records from the file will be forwarded to the Project Manager for inclusion in the project file, or will be placed in bankers boxes (or equivalent) for storage. The final records for storage shall include the following minimum information on placards placed on both the top and end of the storage box:

Database Record File
PROJECT NUMBER: ____
SITE NAME: _____
DATE FILED: __/__/__
SUMMARY OF CONTENTS ENCLOSED
BOX _ OF _

Project- or program-specific record keeping requirements shall take precedence over the record keeping requirements of this SOP.

ATTACHMENT A

MIS REQUEST FORM

Tetra Tech NUS, Inc.

Project Name: | Request Date:
CTO: | Date Data Available for Production:
Project Manager: | Request in Support of:
Requestor: | Database Lead:
Program/Client: | GIS Lead:
State/EPA Region: | Statistics Lead:
| Risk Lead:
Site Name(s) (Area, OU, etc.):
Sampling Date(s):
Matrix: ☐ GW ☐ SO ☐ SD ☐ SW ☐ Other:

Labels: ☐ Labels needed for an upcoming sampling event _____ Total # of Samples
_____ Estimated Hours
_____ Due Date
_____ Complete ETS Charge No.
_____ FOL
Additional Instructions:

Data Entry:
☐ Chemical data needs to be entered from hardcopy _____ Estimated # of Samples
☐ Chemical data needs to be formated electronically
☐ Field analytical data needs to be entered from hardcopy
☐ Geologic data needs to be entered from hardcopy
☐ Hydrology data needs to be entered from hardcopy
_____ Estimated Hours
_____ Due Date
_____ Complete ETS Charge No.
Additional Instructions:

Tables:
☐ Full Data Printout
☐ Summary of Positive Hits
☐ Occurance and Distribution ☐ with criteria
☐ Sampling Analytical Summary
☐ Other:
_____ Estimated Hours
_____ Due Date
_____ Complete ETS Charge No.
Additional Instructions:

GIS:
☐ General Facility Location
☐ Site Location
☐ Potentiometric Contours/Groundwater Flow
☐ Sample Location Proposed
☐ Sample Location Existing
☐ Tag Map Single Round
☐ Tag Map Multiple Round
☐ Isoconcentrations
☐ Chart Map
☐ 3D Visualization
☐ EGIS CD
☐ Other:
_____ Estimated Hours
_____ Due Date
_____ Complete ETS Charge No.
Additional Instructions:

Statistics: ☐ Yes
_____ Estimated Hours
_____ Due Date
_____ Complete ETS Charge No.
Additional Instructions:

Geostatistics: ☐ Yes
_____ Estimated Hours
_____ Due Date
_____ Complete ETS Charge No.
Additional Instructions:

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		Applicability Tetra Tech NUS, Inc.	
		Prepared Earth Sciences Department	
Subject NON-RADIOLOGICAL SAMPLE HANDLING		Approved D. Senovich	

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1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide information on sample preservation, packaging, and shipping procedures to be used in handling environmental samples submitted for chemical constituent, biological, or geotechnical analysis. Sample chain-of-custody procedures and other aspects of field documentation are addressed in SOP SA-6.3. Sample identification is addressed in SOP CT-04.

2.0 SCOPE

This procedure describes the appropriate containers to be used for samples depending on the analyses to be performed, and the steps necessary to preserve the samples when shipped off site for chemical analysis.

3.0 GLOSSARY

Hazardous Material - A substance or material which has been determined by the Secretary of Transportation to be capable of posing an unreasonable risk to health, safety, and property when transported in commerce, and which has been so designated. Under 49 CFR, the term includes hazardous substances, hazardous wastes, marine pollutants, and elevated temperature materials, as well as materials designated as hazardous under the provisions of §172.101 and §172.102 and materials that meet the defining criteria for hazard classes and divisions in Part 173. With slight modifications, IATA has adopted DOT "hazardous materials" as IATA "Dangerous Goods."

Hazardous Waste - Any substance listed in 40 CFR, Subpart D (ỿ261.30 et seq.), or otherwise characterized as ignitable, corrosive, reactive, or toxic (as defined by Toxicity Characteristic Leaching Procedure, TCLP, analysis) as specified under 40 CFR, Subpart C (ỿ261.20 et seq.), that would be subject to manifest requirements specified in 40 CFR 262. Such substances are defined and regulated by EPA.

Marking - A descriptive name, identification number, instructions, cautions, weight, specification or UN marks, or combination thereof required on outer packaging of hazardous materials.

n.o.i - Not otherwise indicated (may be used interchangeably with n.o.s.).

n.o.s. - Not otherwise specified.

Packaging - A receptacle and any other components or materials necessary for compliance with the minimum packaging requirements of 49 CFR 174, including containers (other than freight containers or overpacks), portable tanks, cargo tanks, tank cars, and multi-unit tank-car tanks to perform a containment function in conformance with the minimum packaging requirements of 49 CFR 173.24(a) & (b).

Placard - Color-coded, pictorial sign which depicts the hazard class symbol and name and which is placed on the side of a vehicle transporting certain hazardous materials.

Common Preservatives:

- Hydrochloric Acid - HCl
- Sulfuric Acid - H_2SO_4
- Nitric Acid - HNO_3
- Sodium Hydroxide - NaOH

Other Preservatives

- Zinc Acetate
- Sodium Thiosulfate - $Na_2S_2O_3$

Normality (N) - Concentration of a solution expressed as equivalent per liter, an equivalent being the amount of a substance containing 1 gram-atom of replaceable hydrogen or its equivalent.

Reportable Quantity (RQ) - For the purposes of this SOP, means the quantity specified in column 3 of the Appendix to DOT 49 CFR §172.101 for any material identified in column 1 of the appendix. A spill greater than the amount specified must be reported to the National Response Center.

Sample - A sample is physical evidence collected from a facility or the environment, which is representative of conditions at the location and time of collection.

4.0 RESPONSIBILITIES

Field Operations Leader - Directly responsible for the bottling, preservation, labeling, packaging, shipping, and custody of samples up to and including release to the shipper.

Field Samplers - Responsible for initiating the Chain-of-Custody Record (per SOP SA-6.3), implementing the packaging and shipping requirements, and maintaining custody of samples until they are relinquished to another custodian or to the shipper.

5.0 PROCEDURES

Sample identification, labeling, documentation, and chain-of-custody are addressed by SOP SA-6.3.

5.1 Sample Containers

Different types of chemicals react differently with sample containers made of various materials. For example, trace metals adsorb more strongly to glass than to plastic, whereas many organic chemicals may dissolve various types of plastic containers. Attachments A and B show proper containers (as well as other information) per 40 CFR 136. In general, the sample container shall allow approximately 5-10 percent air space ("ullage") to allow for expansion/vaporization if the sample warms during transport. However, for collection of volatile organic compounds, head space shall be omitted. The analytical laboratory will generally provide certified-clean containers for samples to be analyzed for chemical constituents. Shelby tubes or other sample containers are generally provided by the driller for samples requiring geotechnical analysis. Sufficient lead time shall be allowed for a delivery of sample container orders. Therefore, it is critical to use the correct container to maintain the integrity of the sample prior to analysis.

Once opened, the container must be used at once for storage of a particular sample. Unused but opened containers are to be considered contaminated and must be discarded. Because of the potential for introduction of contamination, they cannot be reclosed and saved for later use. Likewise, any unused containers which appear contaminated upon receipt, or which are found to have loose caps or a missing Teflon liner (if required for the container), shall be discarded.

5.2 Sample Preservation

Many water and soil samples are unstable and therefore require preservation to prevent changes in either the concentration or the physical condition of the constituent(s) requiring analysis. Although complete and irreversible preservation of samples is not possible, preservation does retard the chemical and biological

changes that inevitably take place after the sample is collected. Preservation techniques are usually limited to pH control, chemical addition(s), and refrigeration/ freezing (certain biological samples only).

5.2.1 Overview

The preservation techniques to be used for various analytes are listed in Attachments A and B. Reagents required for sample preservation will either be added to the sample containers by the laboratory prior to their shipment to the field or be added in the field (in a clean environment). Only high purity reagents shall be used for preservation. In general, aqueous samples of low-concentration organics (or soil samples of low- or medium-concentration organics) are cooled to 4°C. Medium-concentration aqueous samples, high-hazard organic samples, and some gas samples are typically not preserved. Low-concentration aqueous samples for metals are acidified with HNO_3, whereas medium-concentration and high-hazard aqueous metal samples are not preserved. Low- or medium-concentration soil samples for metals are cooled to 4°C, whereas high-hazard samples are not cooled.

The following subsections describe the procedures for preparing and adding chemical preservatives. Attachments A and B indicate the specific analytes which require these preservatives.

The FOL is responsible for ensuring that an accurate Chemical Inventory is created and maintained for all hazardous chemicals brought to the work site (see Section 5 of the TtNUS Health and Safety Guidance Manual). Furthermore, the FOL must ensure that a corresponding Material Safety Data Sheet (MSDS) is collected for every substance entered on the site Chemical Inventory, and that all persons using/handling/ disposing of these substances review the appropriate MSDS for substances they will work with. The Chemical Inventory and the MSDSs must be maintained at each work site in a location and manner where they are readily-accessible to all personnel.

5.2.2 Preparation and Addition of Reagents

Addition of the following acids or bases may be specified for sample preservation; these reagents shall be analytical reagent (AR) grade or purer and shall be diluted to the required concentration with deionized water before field sampling commences. To avoid uncontrolled reactions, be sure to Add Acid to water (not vice versa). A dilutions guide is provided below.

Acid/Base	Dilution	Concentration	Estimated Amount Required for Preservation
Hydrochloric Acid (HCl)	1 part concentrated HCl: 1 part double-distilled, deionized water	6N	5-10 mL
Sulfuric Acid (H_2SO_4)	1 part concentrated H_2SO_4: 1 part double-distilled, deionized water	18N	2 - 5 mL
Nitric Acid (HNO_3)	Undiluted concentrated HNO_3	16N	2 - 5 mL
Sodium Hydroxide (NaOH)	400 grams solid NaOH dissolved in 870 mL double-distilled, deionized water; yields 1 liter of solution	10N	2 mL

The amounts required for preservation shown in the above table assumes proper preparation of the preservative and addition of the preservative to one liter of aqueous sample. This assumes that the sample is initially at pH 7, is poorly buffered, and does not contain particulate matter; as these conditions vary, more preservative may be required. Consequently, the final sample pH must be checked using narrow-range pH paper, as described in the generalized procedure detailed below:

- Pour off 5-10 mL of sample into a dedicated, clean container. Use some of this sample to check the initial sample pH using wide range (0-14) pH paper. Never dip the pH paper into the sample; always apply a drop of sample to the pH paper using a clean stirring rod or pipette.

- Add about one-half of the estimated preservative required to the original sample bottle. Cap and invert gently several times to mix. Check pH (as described above) using medium range pH paper (pH 0-6 or pH 7.5-14, as applicable).

- Cap sample bottle and seal securely.

Additional considerations are discussed below:

- To test if ascorbic acid must be used to remove oxidizing agents present in the sample before it can be properly preserved, place a drop of sample on KI-starch paper. A blue color indicates the need for ascorbic acid addition.

  If required, add a few crystals of ascorbic acid to the sample and retest with the KI-starch paper. Repeat until a drop of sample produces no color on the KI-starch paper. Then add an additional 0.6 grams of ascorbic acid per each liter of sample volume.

  Continue with proper base preservation of the sample as described above.

- Samples for sulfide analysis must be treated by the addition of 4 drops (0.2 mL) of 2N zinc acetate solution per 100 ml of sample.

  The 2N zinc acetate solution is made by dissolving 220 grams of zinc acetate in 870 mL of double-distilled, deionized water to make 1 liter of solution.

  The sample pH is then raised to 9 using the NaOH preservative.

- Sodium thiosulfate must be added to remove residual chlorine from a sample. To test the sample for residual chlorine use a field test kit specially made for this purpose.

  If residual chlorine is present, add 0.08 grams of sodium thiosulfate per liter of sample to remove the residual chlorine.

  Continue with proper acidification of the sample as described above.

For biological samples, 10% buffered formalin or isopropanol may also be required for preservation. Questions regarding preservation requirements should be resolved through communication with the laboratory before sampling begins.

5.3 Field Filtration

At times, field-filtration may be required to provide for the analysis of dissolved chemical constituents. Field-filtration must be performed prior to the preservation of samples as described above. General procedures for field filtration are described below:

- The sample shall be filtered through a non-metallic, 0.45-micron membrane filter, immediately after collection. The filtration system shall consist of dedicated filter canister, dedicated tubing, and a peristaltic pump with pressure or vacuum pumping squeeze action (since the sample is filtered by mechanical peristalsis, the sample travels only through the tubing).

- To perform filtration, thread the tubing through the peristaltic pump head. Attach the filter canister to the discharge end of the silicon tubing (note flow direction arrow); attach the aqueous sample container to the intake end of the silicon tubing. Turn the peristaltic pump on and perform filtration. Run approximately 100 ml of sample through the filter and discard prior to sample collection.

- Continue by preserving the filtrate (contained in the filter canister), as applicable and generally described above.

5.4 Sample Packaging and Shipping

Only employees who have successfully completed the TtNUS "Shipping Hazardous Materials" training course are authorized to package and ship hazardous substances. These trained individuals are responsible for performing shipping duties in accordance with this training.

Samples collected for shipment from a site shall be classified as either environmental or hazardous material samples. Samples from drums containing materials other than Investigative Derived Waste (IDW) and samples obtained from waste piles or bulk storage tanks are generally shipped as hazardous materials. A distinction must be made between the two types of samples in order to:

- Determine appropriate procedures for transportation of samples (if there is any doubt, a sample shall be considered hazardous and shipped accordingly.)

- Protect the health and safety of transport and laboratory personnel receiving the samples (special precautions are used by the shipper and at laboratories when hazardous materials are received.)

Detailed procedures for packaging environmental samples are outlined in the remainder of this section.

5.4.1 Environmental Samples

Environmental samples are packaged as follows:

- Place properly identified sample container, with lid securely fastened, in a plastic bag (e.g. Ziploc baggie), and seal the bag.

- Place sample in a cooler constructed of sturdy material which has been lined with a large, plastic bag (e.g. "garbage" bag). Drain plugs on coolers must be taped shut.

- Pack with enough cushioning materials such as bubble wrap (shoulders of bottles must be iced if required) to minimize the possibility of the container breaking.

- If cooling is required (see Attachments A and B), place ice around sample container shoulders, and on top of packing material (minimum of 8 pounds of ice for a medium-size cooler).

- Seal (i.e., tape or tie top in knot) large liner bag.

- The original (top, signed copy) of the COC form shall be placed inside a large Ziploc-type bag and taped inside the lid of the shipping cooler. If multiple coolers are sent but are included on one COC form, the COC form should be sent with the cooler containing the vials for VOC analysis. The COC form should then state how many coolers are included with that shipment.

- Close and seal outside of cooler as described in SOP SA-6.3. Signed custody seals must be used.

Coolers must be marked as containing "Environmental Samples." The appropriate side of the container must be marked "This End Up" and arrows placed appropriately. No DOT marking or labeling is required; there are no DOT restrictions on mode of transportation.

6.0 REFERENCES

American Public Health Association, 1981. Standard Methods for the Examination of Water and Wastewater, 15th Edition. APHA, Washington, D.C.

International Air Transport Association (latest issue). Dangerous Goods Regulations, Montreal, Quebec, Canada.

U.S. Department of Transportation (latest issue). Hazardous Materials Regulations, 49 CFR 171-177.

U.S. EPA, 1984. "Guidelines Establishing Test Procedures for the Analysis of Pollutants under Clean Water Act." Federal Register, Volume 49 (209), October 26, 1984, p. 43234.

U.S. EPA, 1979. Methods for Chemical Analysis of Water and Wastes. EPA-600/4-79-020, U.S. EPA-EMSL, Cincinnati, Ohio.

ATTACHMENT A

GENERAL SAMPLE CONTAINER AND PRESERVATION REQUIREMENTS

Sample Type and Concentration			Container[1]	Sample Size	Preservation[2]	Holding Time[2]
WATER						
Organics (GC&GC/MS)	VOC	Low	Borosilicate glass	2 x 40 mL	Cool to 4°C HCl to ≤ 2	14 days[9]
	Extractables SVOCs and pesticide/PCBs)	(Low	Amber glass	2x2 L or 4x1 L	Cool to 4°C	7 days to extraction; 40 days after extraction
	Extractables SVOCs and pesticide/PCBs)	(Medium	Amber glass	2x2 L or 4x1 L	None	7 days to extraction; 40 days after extraction
Inorganics	Metals	Low	High-density polyethylene	1 L	HNO_3 to pH ≤2	6 months (Hg-28 days)
		Medium	Wide-mouth glass	16 oz.	None	6 months
	Cyanide	Low	High-density polyethylene	1 L	NaOH to pH>12	14 days
	Cyanide	Medium	Wide-mouth glass	16 oz.	None	14 days
Organic/ Inorganic	High Hazard		Wide-mouth glass	8 oz.	None	14 days
SOIL						
Organics (GC&GC/MS)	VOC		EnCore Sampler	(3) 5 g Samplers	Cool to 4°C	48 hours to lab preservation
	Extractables SVOCs and pesticides/PCBs)	(Low	Wide-mouth glass	8 oz.	Cool to 4°C	14 days to extraction; 40 days after extraction
	Extractables SVOCs and pesticides/PCBs)	(Medium	Wide-mouth glass	8 oz.	Cool to 4°C	14 days to extraction; 40 days after extraction
Inorganics	Low/Medium		Wide-mouth glass	8 oz.	Cool to 4°C	6 months (Hg - 28 days) Cyanide (14 days)
Organic/Inorganic	High Hazard		Wide-mouth glass	8 oz.	None	NA
Dioxin/Furan	All		Wide-mouth glass	4 oz.	None	35 days until extraction; 40 days after extraction
TCLP	All		Wide-mouth glass	8 oz.	None	7 days until preparation; analysis as per fraction
AIR						
Volatile Organics	Low/Medium		Charcoal tube -- 7 cm long, 6 mm OD, 4 mm ID	100 L air	Cool to 4°C	5 days recommended

1 All glass containers should have Teflon cap liners or septa.
2 See Attachment E. Preservation and maximum holding time allowances per 40 CFR 136.

ATTACHMENT B

ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES

Parameter Number/Name	Container[1]	Preservation[2][3]	Maximum Holding Time[4]
INORGANIC TESTS:			
Acidity	P, G	Cool, 4°C	14 days
Alkalinity	P, G	Cool, 4°C	14 days
Ammonia - Nitrogen	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Biochemical Oxygen Demand (BOD)	P, G	Cool, 4°C	48 hours
Bromide	P, G	None required	28 days
Chemical Oxygen Demand (COD)	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Chloride	P, G	None required	28 days
Chlorine, Total Residual	P, G	None required	Analyze immediately
Color	P, G	Cool, 4°C	48 hours
Cyanide, Total and Amenable to Chlorination	P, G	Cool, 4°C; NaOH to pH 12; 0.6 g ascorbic acid[5]	14 days[6]
Fluoride	P	None required	28 days
Hardness	P, G	HNO_3 to pH 2; H_2SO_4 to pH 2	6 months
Total Kjeldahl and Organic Nitrogen	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Nitrate - Nitrogen	P, G	None required	48 hours
Nitrate-Nitrite - Nitrogen	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Nitrite - Nitrogen	P, G	Cool, 4°C	48 hours
Oil & Grease	G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Total Organic Carbon (TOC)	P, G	Cool, 4°C; HCl or H_2SO_4 to pH 2	28 days
Orthophosphate	P, G	Filter immediately; Cool, 4°C	48 hours
Oxygen, Dissolved-Probe	G Bottle & top	None required	Analyze immediately
Oxygen, Dissolved-Winkler	G Bottle & top	Fix on site and store in dark	8 hours
Phenols	G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Phosphorus, Total	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Residue, Total	P, G	Cool, 4°C	7 days
Residue, Filterable (TDS)	P, G	Cool, 4°C	7 days
Residue, Nonfilterable (TSS)	P, G	Cool, 4°C	7 days
Residue, Settleable	P, G	Cool, 4°C	48 hours
Residue, Volatile (Ash Content)	P, G	Cool, 4°C	7 days
Silica	P	Cool, 4°C	28 days
Specific Conductance	P, G	Cool, 4°C	28 days
Sulfate	P, G	Cool, 4°C	28 days

ATTACHMENT B
ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES
PAGE TWO

Parameter Number/Name	Container[1]	Preservation[2][3]	Maximum Holding Time[4]
INORGANIC TESTS (Cont'd):			
Sulfide	P, G	Cool, 4°C; add zinc acetate plus sodium hydroxide to pH 9	7 days
Sulfite	P, G	None required	Analyze immediately
Turbidity	P, G	Cool, 4°C	48 hours
METALS:[7]			
Chromium VI (Hexachrome)	P, G	Cool, 4°C	24 hours
Mercury (Hg)	P, G	HNO_3 to pH 2	28 days
Metals, except Chromium VI and Mercury	P, G	HNO_3 to pH 2	6 months
ORGANIC TESTS:[8]			
Purgeable Halocarbons	G, Teflon-lined septum	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	14 days
Purgeable Aromatic Hydrocarbons	G, Teflon-lined septum	Cool, 4°C; 0.008% $Na_2S_2O_3$[5] HCl to pH 2 [9]	14 days
Acrolein and Acrylonitrile	G, Teflon-lined septum	Cool, 4°C; 0.008% $Na_2S_2O_3$[5] adjust pH to 4-5 [10]	14 days
Phenols[11]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction
Benzidines[11],[12]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	7 days until extraction[13]
Phthalate esters[11]	G, Teflon-lined cap	Cool, 4°C	7 days until extraction; 40 days after extraction
Nitrosamines[11],[14]	G, Teflon-lined cap	Cool, 4°C; store in dark; 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction
PCBs[11]	G, Teflon-lined cap	Cool, 4°C	7 days until extraction; 40 days after extraction
Nitroaromatics & Isophorone[11]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]; store in dark	7 days until extraction; 40 days after extraction
Polynuclear Aromatic Hydrocarbons (PAHs)[11],[14]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]; store in dark	7 days until extraction; 40 days after extraction
Haloethers[11]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction
Dioxin/Furan (TCDD/TCDF)[11]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction

ATTACHMENT B
ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES
PAGE THREE

(1) Polyethylene (P): generally 500 ml or Glass (G): generally 1L.
(2) Sample preservation should be performed immediately upon sample collection. For composite chemical samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.
(3) When any sample is to be shipped by common carrier or sent through the United States Mail, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172).
(4) Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that the specific types of samples under study are stable for the longer periods, and has received a variance from the Regional Administrator.
(5) Should only be used in the presence of residual chlorine.
(6) Maximum holding time is 24 hours when sulfide is present. Optionally, all samples may be tested with lead acetate paper before pH adjustments are made to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.
(7) Samples should be filtered immediately on site before adding preservative for dissolved metals.
(8) Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.
(9) Sample receiving no pH adjustment must be analyzed within 7 days of sampling.
(10) The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.
(11) When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6-9; samples preserved in this manner may be held for 7 days before extraction and for 40 days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (re: the requirement for thiosulfate reduction of residual chlorine) and footnotes 12, 13 (re: the analysis of benzidine).
(12) If 1,2-diphenylthydrazine is likely to be present, adjust the pH of the sample to 4.0±0.2 to prevent rearrangement to benzidine.
(13) Extracts may be stored up to 7 days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.
(14) For the analysis of diphenylnitrosamine, add 0.008% $Na_2S_2O_3$ and adjust pH to 7-10 with NaOH within 24 hours of sampling.
(15) The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% $Na_2S_2O_3$.

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		Effective Date 03/09/09	Revision 3
		Applicability Tetra Tech NUS, Inc.	
		Prepared Earth Sciences Department	
Subject FIELD DOCUMENTATION		Approved Tom Johnston	

TABLE OF CONTENTS

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to identify and designate the field data record forms, logs, and reports generally initiated and maintained for documenting Tetra Tech NUS, Inc. (TtNUS) field activities.

2.0 SCOPE

Documents presented within this SOP (or equivalents) shall be used for all TtNUS field activities, as applicable. Other or additional documents may be required by specific client contracts or project planning documents.

3.0 GLOSSARY

None.

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Project Manager (PM) - The PM is responsible for obtaining hardbound controlled-distribution logbooks (from the appropriate source), as needed. In addition, the Project Manager is responsible for placing all field documentation used in site activities (i.e., records, field reports, sample data sheets, field notebooks, and the site logbook) in the project's central file upon the completion of field work.

Field Operations Leader (FOL) - The FOL is responsible for ensuring that the site logbook, notebooks, and all appropriate and current forms and field reports included in this SOP (and any additional forms required by the contract) are correctly used, accurately filled out, and completed in the required time frame.

General personnel qualifications for field documentation activities include the following:

- Occupational Safety and Health Administration (OSHA) 40-hour and applicable refresher training.
- Capability of performing field work under the expected physical and environmental (i.e., weather) conditions.
- Familiarity with appropriate procedures for documentation, handling, packaging, and shipping.

5.0 PROCEDURES

5.1 SITE LOGBOOK

5.1.1 General

The site logbook is a hard-bound, paginated, controlled-distribution record book in which all major on-site activities are documented. At a minimum, record or reference the following activities/events (daily) in the site logbook:

- All field personnel present
- Arrival/departure times and names of site visitors
- Times and dates of health and safety training
- Arrival/departure times of equipment
- Times and dates of equipment calibration

- Start and/or completion of borehole, trench, monitoring well installation activities, etc.
- Daily on-site activities
- Sample pickup information
- Health and safety issues (level of protection, personal protective equipment [PPE], etc.)
- Weather conditions

Maintain a site logbook for each project and initiate it at the start of the first on-site activity (e.g., site visit or initial reconnaissance survey). Make entries every day that on-site activities take place involving TtNUS or subcontractor personnel. Upon completion of the fieldwork, provide the site logbook to the PM or designee for inclusion in the project's central file.

Record the following information on the cover of each site logbook:

- Project name
- TtNUS project number
- Sequential book number
- Start date
- End date

Information recorded daily in the site logbook need not be duplicated in other field notebooks (see Section 5.2) but must summarize the contents of these other notebooks and refer to specific page locations in these notebooks for detailed information (where applicable). An example of a typical site logbook entry is shown in Attachment A.

If measurements are made at any location, either record the measurements and equipment used in the site logbook or reference the field notebook in which the measurements are recorded (see Attachment A).

Make all logbook, notebook, and log sheet entries in indelible ink (black pen is preferred). No erasures are permitted. If an incorrect entry is made, cross out the entry with a single strike mark, initial, and date it. At the completion of entries by any individual, the logbook pages used must be signed and dated by the person making the entries. The site logbook must also be signed by the FOL at the end of each day.

5.1.2 Photographs

Sequentially number movies, slides, or photographs taken of a site or any monitoring location to correspond to logbook/notebook entries. Enter the name of the photographer, date, time, site location, site description, and weather conditions in the logbook/notebook as the photographs are taken. A series entry may be used for rapid-sequence photographs. The photographer is not required to record the aperture settings and shutter speeds for photographs taken within the normal automatic exposure range. However, special lenses, films, filters, and other image-enhancement techniques must be noted in the logbook/notebook. If possible, such techniques shall be avoided because they can adversely affect the accuracy of photographs. Chain-of-custody procedures depend on the subject matter, type of camera (digital or film), and the processing it requires. Follow chain-of-custody procedures for film used for aerial photography, confidential information, or criminal investigation. After processed, consecutively number the slides of photographic prints and label them according to the logbook/notebook descriptions. Docket the site photographs and associated negatives and/or digitally saved images to compact disks into the project's central file.

5.2 FIELD NOTEBOOKS

Key field team personnel may maintain a separate dedicated field notebook to document the pertinent field activities conducted directly under their supervision. For example, on large projects with multiple investigative sites and varying operating conditions, the Health and Safety Officer may elect to maintain a

separate field notebook. Where several drill rigs are in operation simultaneously, each site geologist assigned to oversee a rig must maintain a field notebook.

5.3 FIELD FORMS

All TtNUS field forms (see list in Section 6.0 of this SOP) can be found on the company's intranet site (http://intranet.ttnus.com) under Field Log Sheets. Forms may be altered or revised for project-specific needs, subject to client approval. Care must be taken to ensure that all essential information can be documented. Guidelines for completing these forms can be found in the related sampling SOPs.

5.3.1 Sample Collection, Labeling, Shipment, Request for Analysis, and Field Test Results

5.3.1.1 Sample Log Sheet

Sample log sheets are used to record specified types of data while sampling. The data recorded on these sheets are useful in describing the sample as well as pointing out any problems, difficulties, or irregularities encountered during sampling. Complete a sample log sheet for each sample obtained, including field quality control (QC) samples.

5.3.1.2 Sample Label

A typical sample label is illustrated in Attachment B. Complete the required information on the adhesive labels and apply them to every sample container. Obtain sample labels from the appropriate program/project source, request that they be electronically generated in house, or request them the laboratory subcontractor.

5.3.1.3 Chain-of-Custody Record

The chain-of-custody record is a multi-part form that is initiated as samples are acquired and accompanies a sample (or group of samples) as they are transferred from person to person. This form must be used as follows for any samples collected for chemical or geotechnical analysis whether the analyses are performed on site or off site:

- Retain one carbonless copy of the completed chain-of custody form in the field.
- Send one copy is sent to the PM (or designee)
- Send the original to the laboratory with the associated samples. Place the original (top, signed copy) of the chain-of custody form inside a large Ziploc®-type bag taped inside the lid of the shipping cooler. If multiple coolers are sent but are included on one chain-of custody form, send the form with the cooler containing vials for volatile organic compound (VOC) analysis or the cooler with the air bill attached. Indicate on the air bill how many coolers are included with that shipment.

An example of a chain-of-custody form is provided as Attachment C. After the samples are received at the laboratory, the sample cooler and contents are checked and any problems are noted on the enclosed chain-of custody form (any discrepancies between the sample labels and chain-of custody form and any other problems that are noted are resolved through communication between the laboratory point-of-contact and the TtNUS PM). The chain-of custody form is signed and copied. The laboratory will retain the copy, and the original becomes part of the samples' corresponding analytical data package.

5.3.1.4 Chain-of-Custody Seal

Attachment D is an example of a custody seal. The custody seal is an adhesive-backed label that is part of a chain-of-custody process and is used to prevent tampering with samples after they have been collected in the field and sealed in coolers for transport to the laboratory. Sign and date custody seals

and affix them across the lid and body of each cooler (front and back) containing environmental samples (see SOP SA-6.1). Obtain custody seals from the laboratory (if available) or purchase them from a supplier.

5.3.1.5 Geochemical Parameters Log Sheets

Complete Field Analytical Log Sheets to record geochemical and/or natural attenuation field test results.

5.3.2 Hydrogeological and Geotechnical Forms

5.3.2.1 Groundwater Level Measurement Sheet

Complete a Groundwater Level Measurement Sheet for each round of water level measurements made at a site.

5.3.2.2 Data Sheet for Pumping Test

During the performance of a pumping test (or an in-situ hydraulic conductivity test), a large amount of data must be recorded, often within a short time period. Use a Pumping Test Data Sheet to facilitate this task by standardizing the data collection format for the pumping well and observation wells, and allowing the time interval for collection to be established in advance.

5.3.2.3 Packer Test Report Form

Complete a Packer Test Report Form for each well at which a packer test is conducted.

5.3.2.4 Boring Log

Complete a Summary Log of Boring, or Boring Log for each soil boring performed to document the materials encountered, operation and driving of casing, and locations/depths of samples collected. In addition, if volatile organics are monitored on cores, samples, cuttings from the borehole, or breathing zone, (using a photoionization detector [PID] or flame ionization detector [FID]), enter these readings on the boring log at the appropriate depth. When they become available, enter the laboratory sample number, concentrations of key contaminants, or other pertinent information in the "Remarks" column. This feature allows direct comparison of contaminant concentrations with soil characteristics.

5.3.2.5 Monitoring Well Construction Details Form

Complete a Monitoring Well Construction Details Form for every monitoring well, piezometer, or temporary well point installed. This form contains specific information on length and type of well riser pipe and screen, backfill, filter pack, annular seal and grout characteristics, and surface seal characteristics. This information is important in evaluating the performance of the monitoring well, particularly in areas where water levels show temporal variation or where there are multiple (immiscible) phases of contaminants. Depending on the type of monitoring well (in overburden or bedrock, stick-up or flush mount), different forms are used.

5.3.2.6 Test Pit Log

When a test pit or trench is constructed for investigative or sampling purposes, a Test Pit Log must be filled out by the responsible field geologist or sampling technician.

5.3.2.7 Miscellaneous Monitoring Well Forms

Miscellaneous monitoring well forms that may be required on a project-specific basis include the Monitoring Well Materials Certificate of Conformance and Monitoring Well Development Record. Use a Monitoring Well Materials Certificate of Conformance to document all materials utilized during each monitoring well installation. Use a Monitoring Well Development Record to document all well development activities.

5.3.2.8 Miscellaneous Field Forms – Quality Assurance and Checklists

Miscellaneous field forms/checklists forms that may be required on a project-specific basis include the following:

- Container Sample and Inspection Sheet – use this form when a container (drum, tank, etc.) is sampled and/or inspected.

- QA Sample Log Sheet – use this form when a QA sample such as an equipment rinsate blank, source blank, etc. is collected.

- Field Task Modification Request (FTMR) – use this form to document deviations from the project planning documents. The FOL is responsible for initiating the FTMRs. Maintain copies of all FTMRs with the on-site planning documents, and place originals in the final evidence file.

- Field Project Daily Activities Checklist and Field Project Pre-Mobilization Checklist – used these during both the planning and field effort to ensure that all necessary tasks are planned for and completed. These two forms are not requirements but are useful tools for most field work.

5.3.3 Equipment Calibration and Maintenance Form

The calibration or standardization of monitoring, measuring, or test equipment is necessary to ensure the proper operation and response of the equipment, to document the accuracy, precision, or sensitivity of the measurements, and determine if correction should be applied to the readings. Some items of equipment require frequent calibration, others infrequent. Some are calibrated by the manufacturer, others by the user.

Each instrument requiring calibration has its own Equipment Calibration Log, which documents that the manufacturer's instructions were followed for calibration of the equipment, including frequency and type of standard or calibration device. Maintain an Equipment Calibration Log for each electronic measuring device used in the field; make entries for each day the equipment is used or in accordance with manufacturer recommendations.

5.4 FIELD REPORTS

The primary means of recording on-site activities is the site logbook. Other field notebooks may also be maintained. These logbooks and notebooks (and supporting forms) contain detailed information required for data interpretation or documentation but are not easily used for tracking and reporting of progress. Furthermore, the field logbook/notebooks remain on site for extended periods of time and are thus not accessible for timely review by project management. Other reports useful for tracking and reporting the progress of field activities are described below.

5.4.1 Daily Activities Report

To provide timely oversight of on-site contractors, complete and submit Daily Activities Reports (DARs) as described below.

5.4.1.1 Description

The DAR documents the activities and progress for each day's field work. Complete this report on a daily basis whenever there are drilling, test pitting, well construction, or other related activities occurring that involve subcontractor personnel. These sheets summarize the work performed and form the basis of payment to subcontractors. The DAR form can be found on the TtNUS intranet site.

5.4.1.2 Responsibilities

It is the responsibility of the rig geologist to complete the DAR and obtain the driller's signature acknowledging that the times and quantities of material entered are correct.

5.4.1.3 Submittal and Approval

At the end of the shift, the rig geologist must submit the DAR to the FOL for review and filing. The Daily Activities Report is not a formal report and thus requires no further approval. The DARs are retained by the FOL for use in preparing the site logbook and in preparing weekly status reports for submission to the PM.

5.4.2 Weekly Status Reports

To facilitate timely review by project management, photocopies of logbook/notebook entries may be made for internal use.

In addition to those described herein, other summary reports may also be contractually required.

All TtNUS field forms can be found on the company's intranet site at http://intranet.ttnus.com under Field Log Sheets.

6.0 LISTING OF FIELD FORMS ON THE TtNUS INTRANET SITE

- Boring Log
- Container Sample and Inspection Sheet
- Daily Activities Checklist
- Daily Activities Record
- Equipment Calibration Log
- Field Task Modification Request
- Field Analytical Log sheet - Geochemical Parameters
- Groundwater Level Measurement Sheet
- Groundwater Sample Log Sheet
- Hydraulic Conductivity Test Data Sheet
- Low Flow Purge Data Sheet
- Bedrock Monitoring Well Construction (Stick Up)
- Bedrock Monitoring Well Construction Flush Mount
- Bedrock Monitoring Well Construction Open Hole
- Confining Layer Monitoring Well Construction
- Monitoring Well Development Record

- Monitoring Well Materials Certificate of Conformance
- Overburden Monitoring Well Construction Flush Mount
- Overburden Monitoring Well Construction Stick Up
- Packer Test Report Form
- Pumping Test Data Sheet
- QA Sample Log Sheet
- Soil/Sediment Sample Log Sheet
- Surface Water Sample Log Sheet
- Test Pit Log
- Field Project Pre-Mobilization Checklist

ATTACHMENT A
TYPICAL SITE LOGBOOK ENTRY

START TIME: _____ DATE: _____

SITE LEADER: _____
PERSONNEL:

TtNUS	DRILLER	SITE VISITORS
_____	_____	_____
_____	_____	_____
_____	_____	_____

WEATHER: Clear, 68°F, 2-5 mph wind from SE

ACTIVITIES:

1. Steam jenney and fire hoses were set up.
2. Drilling activities at well ____ resumes. Rig geologist was _____. See Geologist's Notebook, No. 1, page 29-30, for details of drilling activity. Sample No. 123-21-S4 collected; see sample logbook, page 42. Drilling activities completed at 11:50 and a 4-inch stainless steel well installed. See Geologist's Notebook, No. 1, page 31, and well construction details for well _____.
3. Drilling rig No. 2 steam-cleaned at decontamination pit. Then set up at location of well _____.
4. Well _____ drilled. Rig geologist was _____. See Geologist's Notebook, No. 2, page ____ for details of drilling activities. Sample numbers 123-22-S1, 123-22-S2, and 123-22-S3 collected; see sample logbook, pages 43, 44, and 45.
5. Well _____ was developed. Seven 55-gallon drums were filled in the flushing stage. The well was then pumped using the pitcher pump for 1 hour. At the end of the hour, water pumped from well was "sand free."
6. EPA remedial project manger arrives on site at 14:25 hours.
7. Large dump truck arrives at 14:45 and is steam-cleaned. Backhoe and dump truck set up over test pit _____.
8. Test pit _____ dug with cuttings placed in dump truck. Rig geologist was _____. See Geologist's Notebook, No. 1, page 32, for details of test pit activities. Test pit subsequently filled. No samples taken for chemical analysis. Due to shallow groundwater table, filling in of test pit ___ resulted in a very soft and wet area. A mound was developed and the area roped off.
9. Express carrier picked up samples (see Sample Logbook, pages 42 through 45) at 17:50 hours. Site activities terminated at 18:22 hours. All personnel off site, gate locked.

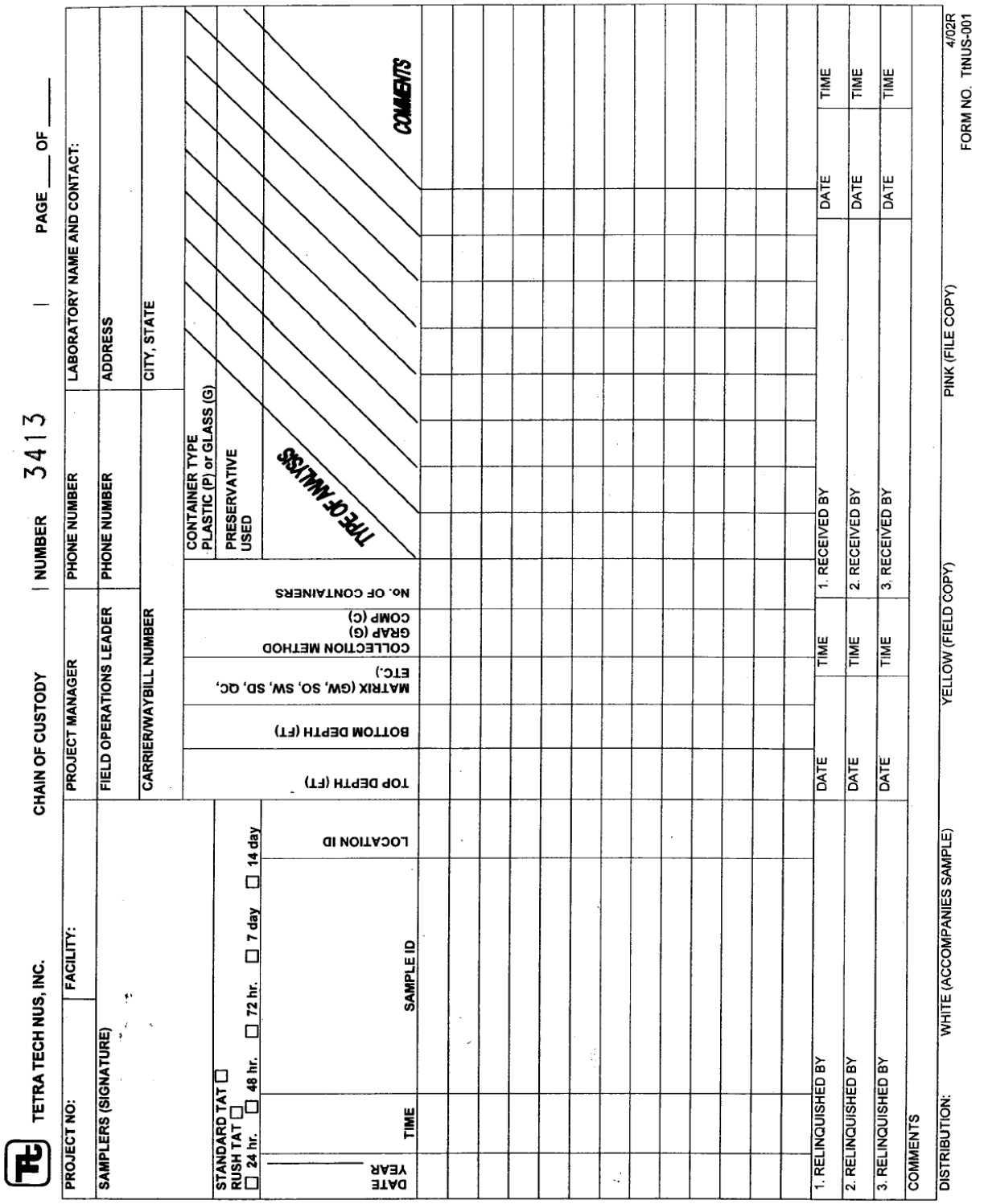
_____

Field Operations Leader

ATTACHMENT B
SAMPLE LABEL

Tetra Tech NUS, Inc. 661 Andersen Drive Pittsburgh, 15220 (412)921-7090		Project: Site: Location:	
Sample No:			Matrix:
Date:	Time:	Preserve:	
Analysis:			
Sampled by:		Laboratory:	

ATTACHMENT C
CHAIN-OF-CUSTODY RECORD FORM



TETRA TECH NUS, INC. CHAIN OF CUSTODY | NUMBER 3413 | PAGE ___ OF ___

PROJECT NO: | FACILITY: | PROJECT MANAGER | PHONE NUMBER | LABORATORY NAME AND CONTACT:

SAMPLERS (SIGNATURE) | FIELD OPERATIONS LEADER | PHONE NUMBER | ADDRESS

CARRIER/WAYBILL NUMBER | CITY, STATE

STANDARD TAT ☐
RUSH TAT ☐ ☐ 24 hr. ☐ 48 hr. ☐ 72 hr. ☐ 7 day ☐ 14 day

DATE / TIME / SAMPLE ID / LOCATION ID / TOP DEPTH (FT) / BOTTOM DEPTH (FT) / MATRIX (GW, SO, SW, SD, QC, ETC.) / COLLECTION METHOD GRAB (G) COMP (C) / No. OF CONTAINERS / CONTAINER TYPE PLASTIC (P) or GLASS (G) / PRESERVATIVE USED / TYPE OF ANALYSIS / COMMENTS

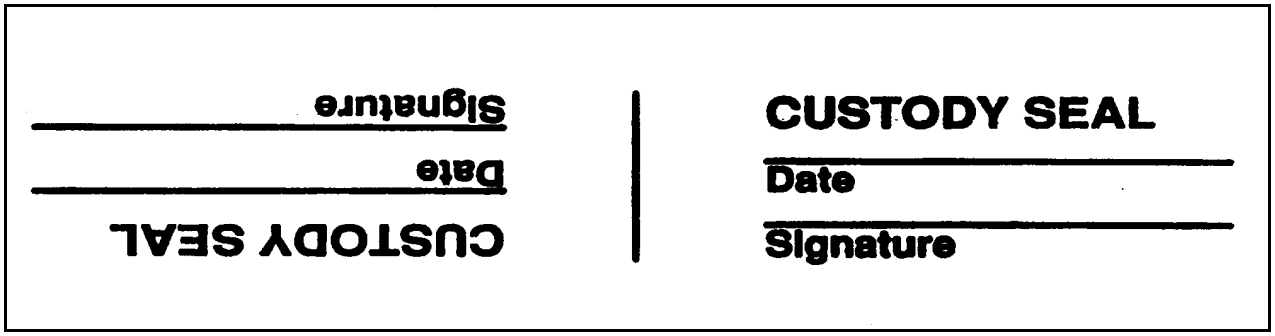
	DATE	TIME		DATE	TIME
1. RELINQUISHED BY			1. RECEIVED BY		
2. RELINQUISHED BY			2. RECEIVED BY		
3. RELINQUISHED BY			3. RECEIVED BY		

COMMENTS

DISTRIBUTION: WHITE (ACCOMPANIES SAMPLE) YELLOW (FIELD COPY) PINK (FILE COPY)

4/02R
FORM NO. TtNUS-001

ATTACHMENT D
CHAIN-OF-CUSTODY SEAL



TETRA TECH NUS, INC.	STANDARD OPERATING PROCEDURES	Number HS-1.0	Page 1 of 15
		Effective Date 12/03	Revision 2
		Applicability Tetra Tech NUS, Inc.	
		Prepared Health & Safety	
Subject UTILITY LOCATING AND EXCAVATION CLEARANCE		Approved D. Senovich	

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1.0 PURPOSE

Utilities such as electric service lines, natural or propane gas lines, water and sewage lines, telecommunications, and steam lines are very often in the immediate vicinity of work locations. Contact with underground or overhead utilities can have serious consequences including employee injury/fatality, property and equipment damage, substantial financial impacts, and loss of utility service to users.

The purpose of this procedure is to provide minimum requirements and technical guidelines regarding the appropriate procedures to be followed when performing subsurface and overhead utility locating services. It is the policy of Tetra Tech NUS, Inc. (TtNUS) to provide a safe and healthful work environment for the protection of our employees. The purpose of this Standard Operating Procedure (SOP) is to aid in achieving the objectives of this policy, to present the acceptable procedures pertaining to utility locating and excavation clearance activities, and to present requirements and restrictions relevant to these types of activities. This SOP must be reviewed by any employee potentially involved with underground or overhead utility locating and avoidance activities.

2.0 SCOPE

This procedure applies to all TtNUS field activities where there may be potential contact with underground or overhead utilities. This procedure provides a description of the principles of operation, instrumentation, applicability, and implementability of typical methods used to determine the presence and avoidance of contact with utility services. This procedure is intended to assist with work planning and scheduling, resource planning, field implementation, and subcontractor procurement. Utility locating and excavation clearance requires site-specific information prior to the initiation of any such activities on a specific project. This SOP is not intended to provide a detailed description of methodology and instrument operation. Specialized expertise during both planning and execution of several of the methods presented may also be required.

3.0 GLOSSARY

Electromagnetic Induction (EMI) Survey - A geophysical exploration method whereby electromagnetic fields are induced in the ground and the resultant secondary electromagnetic fields are detected as a measure of ground conductivity.

Magnetometer – A device used for precise and sensitive measurements of magnetic fields.

Magnetic Survey – A geophysical survey method that depends on detection of magnetic anomalies caused by the presence of buried ferromagnetic objects.

Metal Detection – A geophysical survey method that is based on electromagnetic coupling caused by underground conductive objects.

Vertical Gradiometer – A magnetometer equipped with two sensors that are vertically separated by a fixed distance. It is best suited to map near surface features and is less susceptible to deep geologic features.

Ground Penetrating Radar – Ground Penetrating Radar (GPR) involves specialized radar equipment whereby a signal is sent into the ground via a transmitter. Some portion of the signal will be reflected from the subsurface material, which is then recorded with a receiver and electronically converted into a graphic picture.

4.0 RESPONSIBILITIES

Project Manager (PM)/Task Order Manager (TOM) - Responsible for ensuring that all field activities are conducted in accordance with this procedure.

Site Manager (SM)/Field Operations Leader (FOL) - Responsible for the onsite verification that all field activities are performed in compliance with approved SOPs or as otherwise directed by the approved project plan(s).

Site Health & Safety Officer (SHSO) – Responsible to provide technical assistance and verify full compliance with this SOP. The SHSO is also responsible for reporting any deficiencies to the Corporate Health and Safety Manager (HSM) and to the PM/TOM.

Health & Safety Manager (HSM) – Responsible for preparing, implementing, and modifying corporate health and safety policy and this SOP.

Site Personnel – Responsible for performing their work activities in accordance with this SOP and the TtNUS Health and Safety Policy.

5.0 PROCEDURES

This procedure addresses the requirements and technical procedures that must be performed to minimize the potential for contact with underground and overhead utility services. These procedures are addressed individually from a buried and overhead standpoint.

5.1 Buried Utilities

Buried utilities present a heightened concern because their location is not typically obvious by visual observation, and it is common that their presence and/or location is unknown or incorrectly known on client properties. This procedure must be followed prior to beginning any subsurface probing or excavation that might potentially be in the vicinity of underground utility services. In addition, the Utility Clearance Form (Attachment 3) must be completed for every location or cluster of locations where intrusive activities will occur.

Where the positive identification and de-energizing of underground utilities cannot be obtained and confirmed using the following steps, the PM/TOM is responsible for arranging for the procurement of a qualified, experienced, utility locating subcontractor who will accomplish the utility location and demarcation duties specified herein.

1. A comprehensive review must be made of any available property maps, blue lines, or as-builts prior to site activities. Interviews with local personnel familiar with the area should be performed to provide additional information concerning the location of potential underground utilities. Information regarding utility locations shall be added to project maps upon completion of this exercise.

2., A visual site inspection must be performed to compare the site plan information to actual field conditions. Any findings must be documented and the site plan/maps revised. The area(s) of proposed excavation or other subsurface activities must be marked at the site in white paint or pin flags to identify those locations of the proposed intrusive activities. The site inspection should focus on locating surface indications of potential underground utilities. Items of interest include the presence of nearby area lights, telephone service, drainage grates, fire hydrants, electrical service vaults/panels, asphalt/concrete scares and patches, and topographical depressions. Note the location of any emergency shut off switches. Any additional information regarding utility

locations shall be added to project maps upon completion of this exercise and returned to the PM/TOM.

3. If the planned work is to be conducted on private property (e.g., military installations, manufacturing facilities, etc.) the FOL must identify and contact appropriate facility personnel (e.g., public works or facility engineering) before any intrusive work begins to inquire about (and comply with) property owner requirements. It is important to note that private property owners may require several days to several weeks advance notice prior to locating utilities.

4. If the work location is on public property, the state agency that performs utility clearances must be notified (see Attachment 1). State "one-call" services must be notified prior to commencing fieldwork per their requirements. Most one-call services require, by law, 48- to 72-hour advance notice prior to beginning any excavation. Such services typically assign a "ticket" number to the particular site. This ticket number must be recorded for future reference and is valid for a specific period of time, but may be extended by contacting the service again. The utility service will notify utility representatives who then mark their respective lines within the specified time frame. It should be noted that most military installations own their own utilities but may lease service and maintenance from area providers. Given this situation, "one call" systems may still be required to provide location services on military installations.

5. Utilities must be identified and their locations plainly marked using pin flags, spray paint, or other accepted means. The location of all utilities must be noted on a field sketch for future inclusion on project maps. Utility locations are to be identified using the following industry-standard color code scheme, unless the property owner or utility locator service uses a different color code:

white	excavation/subsurface investigation location
red	electrical
yellow	gas, oil, steam
orange	telephone, communications
blue	water, irrigation, slurry
green	sewer, drain

6. Where utility locations are not confirmed with a high degree of confidence through drawings, schematics, location services, etc., the work area must be thoroughly investigated prior to beginning the excavation. In these situations, utilities must be identified using safe and effective methods such as passive and intrusive surveys, or the use of non-conductive hand tools. Also, in situations where such hand tools are used, they should always be used in conjunction with suitable detection equipment, such as the items described in Section 6.0 of this SOP. Each method has advantages and disadvantages including complexity, applicability, and price. It also should be noted that in some states, initial excavation is required by hand to a specified depth.

7. At each location where trenching or excavating will occur using a backhoe or other heavy equipment, and where utility identifications and locations cannot be confirmed prior to groundbreaking, the soil must be probed using a device such as a tile probe which is made of non-conductive material such as fiberglass. If these efforts are not successful in clearing the excavation area of suspect utilities, hand shoveling must be performed for the perimeter of the intended excavation.

8. All utilities uncovered or undermined during excavation must be structurally supported to prevent potential damage. Unless necessary as an emergency corrective measure, TtNUS shall not make any repairs or modifications to existing utility lines without prior permission of the utility owner, property owner, and Corporate HSM. All repairs require that the line be locked-out/tagged-out prior to work.

5.2 Overhead Power Lines

If it is necessary to work within the minimum clearance distance of an overhead power line, the overhead line must be de-energized and grounded, or re-routed by the utility company or a registered electrician. If protective measures such as guarding, isolating, or insulating are provided, these precautions must be adequate to prevent employees from contacting such lines directly with any part of their body or indirectly though conductive materials, tools, or equipment.

The following table provides the required minimum clearances for working in proximity to overhead power lines.

Nominal Voltage	Minimum Clearance
0 -50 kV	10 feet, or one mast length; whichever is greater
50+ kV	10 feet plus 4 inches for every 10 kV over 50 kV or 1.5 mast lengths; whichever is greater

6.0 UNDERGROUND LOCATING TECHNIQUES

A variety of supplemental utility locating approaches are available and can be applied when additional assurance is needed. The selection of the appropriate method(s) to employ is site-specific and should be tailored to the anticipated conditions, site and project constraints, and personnel capabilities.

6.1 Geophysical Methods

Geophysical methods include electromagnetic induction, magnetics, and ground penetrating radar. Additional details concerning the design and implementation of electromagnetic induction, magnetics, and ground penetrating radar surveys can be found in one or more of the TtNUS SOPs included in the References (Section 8.0).

Electromagnetic Induction

Electromagnetic Induction (EMI) line locators operate either by locating a background signal or by locating a signal introduced into the utility line using a transmitter. A utility line acts like a radio antenna, producing electrons, which can be picked up with a radiofrequency receiver. Electrical current carrying conductors have a 60HZ signal associated with them. This signal occurs in all power lines regardless of voltage. Utilities in close proximity to power lines or used as grounds may also have a 60HZ signal, which can be picked up with an EM receiver. A typical example of this type of geophysical equipment is an EM-61.

EMI locators specifically designed for utility locating use a special signal that is either indirectly induced onto a utility line by placing the transmitter above the line or directly induced using an induction clamp. The clamp induces a signal on the specific utility and is the preferred method of tracing since there is little chance of the resulting signals being interfered with. A good example of this type of equipment is the Schonstedt® MAC-51B locator. The MAC-51B performs inductively traced surveys, simple magnetic locating, and traced nonmetallic surveys.

When access can be gained inside a conduit to be traced, a flexible insulated trace wire can be used. This is very useful for non-metallic conduits but is limited by the availability of gaining access inside the pipe.

Magnetics

Magnetic locators operate by detecting the relative amounts of buried ferrous metal. They are incapable of locating or identifying nonferrous utility lines but can be very useful for locating underground storage tanks (UST's), steel utility lines, and buried electrical lines. A typical example of this type of equipment is the Schonstedt® GA-52Cx locator. The GA-52Cx is capable of locating 4-inch steel pipe up to 8 feet deep.

Non-ferrous lines are often located by using a typical plumbing tool (snake) fed through the line. A signal is then introduced to the snake that is then traced.

Ground Penetrating Radar

Ground Penetrating Radar (GPR) involves specialized radar equipment whereby a signal is sent into the ground via a transmitter. Some portion of the signal will be reflected from the subsurface material, which is then recorded with a receiver and electronically converted into a graphic picture. In general, an object which is harder than the surrounding soil will reflect a stronger signal. Utilities, tunnels, UST's, and footings will reflect a stronger signal than the surrounding soil. Although this surface detection method may determine the location of a utility, this method does not specifically identify utilities (i.e., water vs. gas, electrical vs. telephone); hence, verification may be necessary using other methods. This method is somewhat limited when used in areas with clay soil types or with a high water table.

6.2 Passive Detection Surveys

Acoustic Surveys

Acoustic location methods are generally most applicable to waterlines or gas lines. A highly sensitive Acoustic Receiver listens for background sounds of water flowing (at joints, leaks, etc.) or to sounds introduced into the water main using a transducer. Acoustics may also be applicable to determine the location of plastic gas lines.

Thermal Imaging

Thermal (i.e., infrared) imaging is a passive method for detecting the heat emitted by an object. Electronics in the infrared camera convert subtle heat differentials into a visual image on the viewfinder or a monitor. The operator does not look for an exact temperature; rather they look for heat anomalies (either elevated or suppressed temperatures) characteristic of a potential utility line.

The thermal fingerprint of underground utilities results from differences in temperature between the atmosphere and the fluid present in a pipe or the heat generated by electrical resistance. In addition, infrared scanners may be capable of detecting differences in the compaction, temperature and moisture content of underground utility trenches. High-performance thermal imagery can detect temperature differences to hundredths of a degree.

6.3 Intrusive Detection Surveys

Vacuum Excavation

Vacuum excavation is used to physically expose utility services. The process involves removing the surface material over approximately a 1' x 1' area at the site location. The air-vacuum process proceeds with the simultaneous action of compressed air-jets to loosen soil and vacuum extraction of the resulting

debris. This process ensures the integrity of the utility line during the excavation process, as no hammers, blades, or heavy mechanical equipment comes into contact with the utility line, eliminating the risk of damage to utilities. The process continues until the utility is uncovered. Vacuum excavation can be used at the proposed site location to excavate below the "utility window" which is usually 8 feet.

Hand Excavation

When the identification and location of underground utilities cannot be positively confirmed through document reviews and/or other methods, borings and excavations may be cleared via the use of non-conductive hand tools. This should always be done in conjunction with the use of detection equipment. This would be required for all locations where there is a potential to impact buried utilities. The minimum hand-excavation depth that must be reached is to be determined considering the geographical location of the work site. This approach recognizes that the placement of buried utilities is influenced by frost line depths that vary by geographical region. Attachment 2 presents frost line depths for the regions of the contiguous United States. At a minimum, hand excavation depths must be at least to the frost line depth (see Attachment 2) plus two (2) feet, but never less than 4 feet below ground surface (bgs). For hand excavation, the hole created must be reamed large enough to be at least the diameter of the drill rig auger or bit prior to drilling. For soil gas surveys, the survey probe shall be placed as close as possible to the cleared hand excavation. It is important to note that a post-hole digger must not be used in this type of hand excavation activity.

Tile Probe Surveys

For some soil types, site conditions, and excavation requirements, non-conductive tile probes may be used. A tile probe is a "T"-handled rod of varying lengths that can be pushed into the soil to determine if any obstructions exist at that location. Tile probes constructed of fiberglass or other nonconductive material are readily-available from numerous vendors. Tile probes must be performed to the same depth requirements as previously specified. As with other types of hand excavating activities, the use of a non-conductive tile probe, should always be in conjunction with suitable utility locating detection equipment.

7.0 INTRUSIVE ACTIVITIES SUMMARY

The following list summarizes the activities that must be performed prior to beginning subsurface activities:

1. Map and mark all subsurface locations and excavation boundaries using white paint or markers specified by the client or property owner.

2. Notify the property owner and/or client that the locations are marked. At this point, drawings of locations or excavation boundaries shall be provided to the property owner and/or client so they may initiate (if applicable) utility clearance.

   Note: Drawings with confirmed locations should be provided to the property owner and/or client as soon as possible to reduce potential time delays.

3. Notify "One Call" service. If possible, arrange for an appointment to show the One Call representative the surface locations or excavation boundaries in person. This will provide a better location designation to the utilities they represent. You should have additional drawings should you need to provide plot plans to the One Call service.

4. Implement supplemental utility detection techniques as necessary and appropriate to conform utility locations or the absence thereof.

5. Complete Attachment 3, Utility Clearance Form. This form should be completed for each excavation location. In situations where multiple subsurface locations exist within the close proximity of one another, one form may be used for multiple locations provided those locations are noted on the Utility Clearance Form. Upon completion, the Utility Clearance Form and revised/annotated utility location map becomes part of the project file.

8.0 REFERENCES

OSHA Letter of Interpretation, Mr. Joseph Caldwell, Attachment 4
OSHA 29 CFR 1926(b)(2)
OSHA 29 CFR 1926(b)(3)
TtNUS Utility Locating and Clearance Policy
TtNUS SOP GH-3.1; Resistivity and Electromagnetic Induction
TtNUS SOP GH-3.2; Magnetic and Metal Detection Surveys
TtNUS SOP GH-3.4; Ground-penetrating Radar Surveys

ATTACHMENT 1
LISTING OF UNDERGROUND UTILITY CLEARANCE RESOURCES

American Public Works Association
2345 Grand Boulevard, Suite 500, Kansas City, MO 64108-2625
Phone (816) 472-6100 • Fax (816) 472-1610
Web www.apwa.net • E-mail apwa@apwa.net

ONE-CALL SYSTEMS INTERNATIONAL CONDENSED DIRECTORY

Alabama
Alabama One-Call
1-800-292-8525

Alaska
Locate Call Center of Alaska, Inc.
1-800-478-3121

Arizona
Arizona Blue Stake
1-800-782-5348

Arkansas
Arkansas One Call System, Inc.
1-800-482-8998

California
Underground Service Alert North
1-800-227-2600
Underground Service Alert of Southern California
1-800-227-2600

Colorado
Utility Notification Center of Colorado
1-800-922-1987

Connecticut
Call Before You Dig
1-800-922-4455

Delaware
Miss Utility of Delmarva
1-800-282-8555

Florida
Sunshine State One-Call of Florida, Inc.
1-800-432-4770

Georgia
Underground Protection Center, Inc.
1-800-282-7411

Hawaii
Underground Service Alert North
1-800-227-2600

Idaho
Dig Line Inc.
1-800-342-1585
Kootenai County One-Call
1-800-428-4950
Shoshone - Benewah One-Call
1-800-398-3285

Illinois
JULIE, Inc.
1-800-892-0123
Digger (Chicago Utility Alert Network)
312-744-7000

Indiana
Indiana Underground Plant Protection Service
1-800-382-5544

Iowa
Iowa One-Call
1-800-292-8989

Kansas
Kansas One-Call System, Inc.
1-800-344-7233

Kentucky
Kentucky Underground Protection Inc.
1-800-752-6007

Louisiana
Louisiana One Call System, Inc.
1-800-272-3020

Maine
Dig Safe System, Inc.
1-888-344-7233

Maryland
Miss Utility
1-800-257-7777
Miss Utility of Delmarva
1-800-282-8555

Massachusetts
Dig Safe System, Inc.
1-888-344-7233

Michigan
Miss Dig System, Inc.
1-800-482-7171

Minnesota
Gopher State One Call
1-800-252-1166

Mississippi
Mississippi One-Call System, Inc
1-800-227-6477

Missouri
Missouri One-Call System, Inc.
1-800-344-7483

Montana
Utilities Underground Protection Center
1-800-424-5555
Montana One Call Center
1-800-551-8344

Nebraska
Diggers Hotline of Nebraska
1-800-331-5666

Nevada
Underground Service Alert North
1-800-227-2600

New Hampshire
Dig Safe System, Inc.
1-888-344-7233

New Jersey
New Jersey One Call
1-800-272-1000

New Mexico
New Mexico One Call System, Inc.
1-800-321-2537
Las Cruces- Dona Ana Blue Stakes
1-888-526-0400

New York
Dig Safely New York
1-800-962-7962
New York City- Long Island One Call Center
1-800-272-4480

North Carolina
The North Carolina One-Call Center, Inc.
1-800-632-4949

North Dakota
North Dakota One-Call
1-800-795-0555

Ohio
Ohio Utilities Protection Service
1-800-362-2764
Oil & Gas Producers Underground Protect'n Svc
1-800-925-0988

Oklahoma
Call Okie
1-800-522-6543

Oregon
Oregon Utility Notification Center/One Call Concepts
1-800-332-2344

Pennsylvania
Pennsylvania One Call System, Inc.
1-800-242-1776

Rhode Island
Dig Safe System, Inc.
1-888-344-7233

South Carolina
Palmetto Utility Protection Service Inc.
1-888-721-7877

South Dakota
South Dakota One Call
1-800-781-7474

Tennessee
Tennessee One-Call System, Inc.
1-800-351-1111

ATTACHMENT 1 (Continued)

Texas
Texas One Call System
1-800-245-4545
Texas Excavation Safety System, Inc.
1-800-344-8377
Lone Star Notification Center
1-800-669-8344

Utah
Blue Stakes of Utah
1-800-662-4111

Vermont
Dig Safe System, Inc.
1-888-344-7233

Virginia
Miss Utility of Virginia
1-800-552-7001
Miss Utility (Northern Virginia)
1-800-257-7777

Washington
Utilities Underground Location Center
1-800-424-5555
Northwest Utility Notification Center
1-800-553-4344
Inland Empire Utility Coordinating Council
509-456-8000

West Virginia
Miss Utility of West Virginia, Inc.
1-800-245-4848

Wisconsin
Diggers Hotline, Inc.
1-800-242-8511

Wyoming
Wyoming One-Call System, Inc.
1-800-348-1030
Call Before You Dig of Wyoming
1-800-849-2476

District of Columbia
Miss Utility
1-800-257-7777

Alberta
Alberta One-Call Corporation
1-800-242-3447

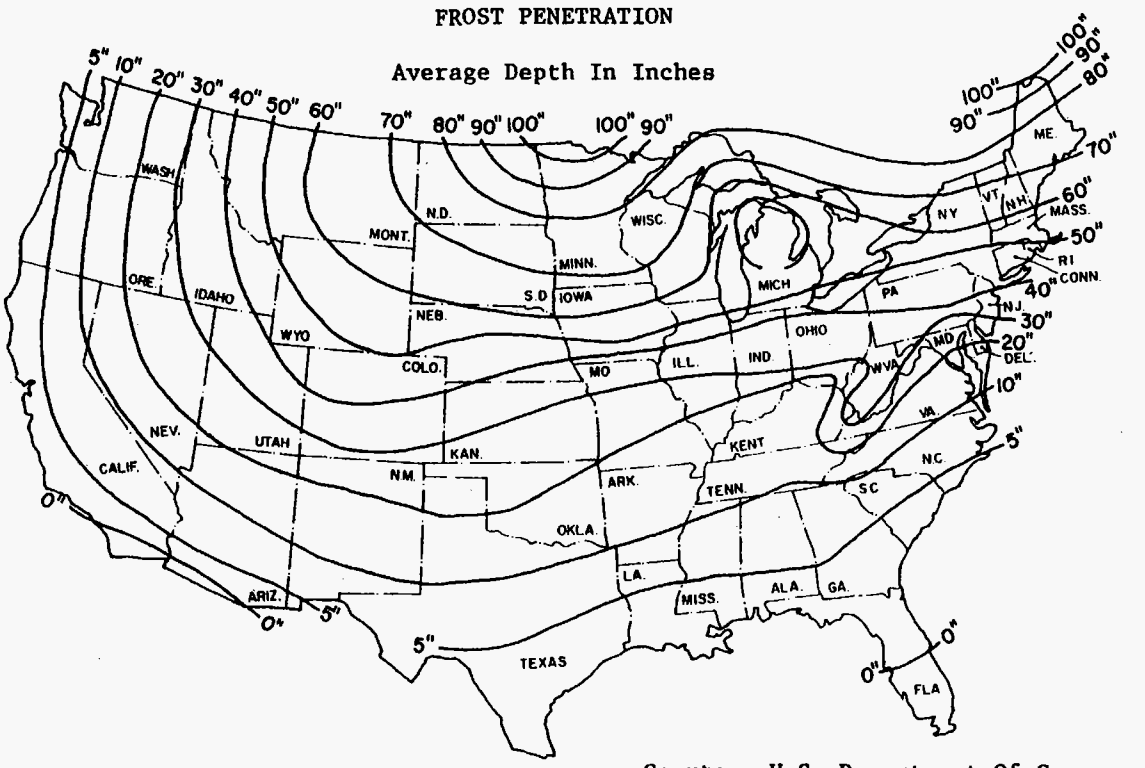
British Columbia
BC One Call
1-800-474-6886

Ontario
Ontario One-Call System
1-800-400-2255

Quebec
Info-Excavation
1-800-663-9228

ATTACHMENT 2

FROST LINE PENETRATION DEPTHS BY GEOGRAPHIC LOCATION



Courtesy U.S. Department Of Commerce

ATTACHMENT 3
UTILITY CLEARANCE FORM

Client: _____ Project Name: _____

Project No.: _____ Completed By: _____

Location Name: _____ Work Date: _____

Excavation Method/Overhead Equipment: _____

			Circle One
1.		Underground Utilities	
	a)	Review of existing maps?	yes no N/A
	b)	Interview local personnel?	yes no N/A
	c)	Site visit and inspection?	yes no N/A
	d)	Excavation areas marked in the field?	yes no N/A
	e)	Utilities located in the field?	yes no N/A
	f)	Located utilities marked/added to site maps?	yes no N/A
	g)	Client contact notified Name _____ Telephone: _____ Date: _____	yes no N/A
	g)	State One-Call agency called? Caller: _____ Ticket Number: _____ Date: _____	yes no N/A
	h)	Geophysical survey performed? Survey performed by: _____ Method: _____ Date: _____	yes no N/A
	i)	Hand excavation performed (with concurrent use of utility detection device)? Completed by: _____ Total depth: _____ feet Date: _____	yes no N/A
	j)	Trench/excavation probed? Probing completed by: _____ Depth/frequency: _____ Date: _____	yes no N/A

			Present Absent
2.		Overhead Utilities	
	a)	Determination of nominal voltage	yes no N/A
	b)	Marked on site maps	yes no N/A
	c)	Necessary to lockout/insulate/re-route	yes no N/A
	d)	Document procedures used to lockout/insulate/re-route	yes no N/A
	e)	Minimum acceptable clearance (SOP Section 5.2): _____	

3. Notes:

_____

_____

_____

Approval:

_____ _____

Site Manager/Field Operations Leader Date

c: PM/Project File
Program File

ATTACHMENT 4
OSHA LETTER OF INTERPRETATION

Mr. Joseph Caldwell
Consultant
Governmental Liaison
Pipeline Safety Regulations
211 Wilson Boulevard
Suite 700
Arlington, Virginia 22201

Re: Use of hydro-vacuum or non-conductive hand tools to locate underground utilities.

Dear Mr. Caldwell:

In a letter dated July 7, 2003, we responded to your inquiry of September 18, 2002, regarding the use of hydro-vacuum equipment to locate underground utilities by excavation. After our letter to you was posted on the OSHA website, we received numerous inquiries that make it apparent that aspects of our July 7 letter are being misunderstood. In addition, a number of industry stakeholders, including the National Utility Contractors Association (NUCA), have provided new information regarding equipment that is available for this work.

To clarify these issues, we are withdrawing our July 7 letter and issuing this replacement response to your inquiry.

Question: *Section 1926.651 contains several requirements that relate to the safety of employees engaged in excavation work. Specifically, paragraphs (b)(2) and (b)(3) relate in part to the safety of the means used to locate underground utility installations that, if damaged during an uncovering operation, could pose serious hazards to employees.*

Under these provisions, what constitutes an acceptable method of uncovering underground utility lines, and further, would the use of hydro-vacuum excavation be acceptable under the standard?

Answer

Background

Two sections of 29 CFR 1926 Subpart P (Excavations), 1926.651(Specific excavation requirements), govern methods for uncovering underground utility installations. Specifically, paragraph (b)(2) states:

> When utility companies or owners cannot respond to a request to locate underground utility installations within 24 hours * * * or cannot establish the exact location of these installations, the employer may proceed, provided the employer does so with caution, and provided detection equipment or other acceptable means to locate utility installations are used. (emphasis added).

Paragraph (b)(3) provides:

ATTACHMENT 4 (Continued)

> When excavation operations approach the estimated location of underground installations, the exact location of the installations shall be determined by safe and acceptable means. (emphasis added).

Therefore, "acceptable means" must be used where the location of the underground utilities have not been identified by the utility companies and detection equipment is not used.

Subpart P does not contain a definition of either "other acceptable means" or "safe and acceptable means." The preambles to both the proposed rule and the final rule discussed the rationale behind the wording at issue. For example, the preamble to the proposed rule, 52 Fed. Reg. 12301 (April 15, 1987), noted that a 1972 version of this standard contained language that specified "careful probing or hand digging" as the means to uncover utilities. The preamble then noted that an amendment to the 1972 standard later deleted that language "to allow other, *equally effective means* of locating such installations." The preamble continued that in the 1987 proposed rule, OSHA again proposed using language in section (b)(3) that would provide another example of an acceptable method of uncovering utilities that could be used *where the utilities have not been marked and detection equipment is not being used* – "probing with hand-held tools." This method was rejected in the final version of 29 CFR 1926. As OSHA explained in the preamble to the final rule, 54 Fed. Reg. 45916 (October 31, 1989):

> OSHA received two comments * * * and input from ACCSH [OSHA's Advisory Committee on Construction Safety and Health] * * * on this provision. All commenters recommended dropping 'such as probing with hand-held tools' from the proposed provision, because this could create a hazard to employees by damaging the installation or its insulation.

In other words, the commenters objected to the use of hand tools being used unless detection equipment was used in conjunction with them. OSHA then concluded its discussion relative to this provision by agreeing with the commentators and ultimately not including any examples of "acceptable means" in the final provision.

Non-conductive hand tools are permitted

This raises the question of whether the standard permits the use of hand tools alone -- without also using detection equipment. NUCA and other industry stakeholders have recently informed us that non-conductive hand tools that are appropriate to be used to locate underground utilities are now commonly available.

Such tools, such as a "shooter" (which has a non-conductive handle and a snub nose) and non-conductive or insulated probes were not discussed in the rulemaking. Since they were not considered at that time, they were not part of the class of equipment that was thought to be unsafe for this purpose. Therefore, we conclude that the use of these types of hand tools, when used with appropriate caution, is an "acceptable means" for locating underground utilities.

ATTACHMENT 4 (Continued)

Hydro-vacuum excavation

It is our understanding that some hydro-vacuum excavation equipment can be adjusted to use a minimum amount of water and suction pressure. When appropriately adjusted so that the equipment will not damage underground utilities (especially utilities that are particularly vulnerable to damage, such as electrical lines), use of such equipment would be considered a "acceptable means" of locating underground utilities. However, if the equipment cannot be sufficiently adjusted, then this method would not be acceptable under the standard.

Other technologies

We are not suggesting that these are the only devices that would be "acceptable means" under the standard. Industry stakeholders have informed us that there are other types of special excavation equipment designed for safely locating utilities as well.

We apologize for any confusion our July 7 letter may have caused. If you have further concerns or questions, please feel free to contact us again by fax at: U.S. Department of Labor, OSHA, Directorate of Construction, Office of Construction Standards and Compliance Assistance, fax # 202-693-1689. You can also contact us by mail at the above office, Room N3468, 200 Constitution Avenue, N.W., Washington, D.C. 20210, although there will be a delay in our receiving correspondence by mail.

Sincerely,

Russell B. Swanson, Director
Directorate of Construction

NOTE: OSHA requirements are set by statute, standards and regulations. Our interpretation letters explain these requirements and how they apply to particular circumstances, but they cannot create additional employer obligations. This letter constitutes OSHA=s interpretation of the requirements discussed. Note that our enforcement guidance may be affected by changes to OSHA rules. Also, from time to time we update our guidance in response to new information. To keep apprised of such developments, you can consult OSHA's website at http://www.osha.gov.

FC 1000. CLEANING / DECONTAMINATION PROCEDURES

1. PERFORMANCE CRITERIA

1.1. The cleaning/decontamination procedures must ensure that all equipment that contacts a sample during sample collection is free from the analytes of interest and constituents that would interfere with the analytes of interest.

1.2. The detergents and other cleaning supplies cannot contribute analytes of interest or interfering constituents unless these are effectively removed during a subsequent step in the cleaning procedure.

1.3. The effectiveness of any cleaning procedure (including all cleaning reagents) must be supported by equipment blanks with reported non-detected values.

The cleaning procedures outlined in this SOP are designed to meet the above-mentioned performance criteria. Alternative cleaning reagents or procedures may be used. However, the organization must be prepared to demonstrate through documentation (i.e., company-written protocols and analytical records) and historical data (i.e., absence of analytes of interest in equipment blanks) that it consistently meets these performance criteria. Field quality control measures (see FQ 1210) must support the use of alternative reagents or procedures.

FC 1001. *Cleaning Reagents*

Recommendations for the types and grades of various cleaning supplies are outlined below. The recommended reagent types or grades were selected to ensure that the cleaned equipment is free from any detectable contamination.

1. DETERGENTS: Use Luminox (or a non-phosphate solvent based equivalent), Liqui-Nox (or a non-phosphate equivalent) or Alconox (or equivalent). EPA recommends Luminox (or equivalent) since solvent rinses can be eliminated from the cleaning process. Liquinox (or equivalent) may be substituted (solvent rinses, when applicable, must be performed), and Alconox (or equivalent) may be substituted if the sampling equipment will not be used to collect phosphorus or phosphorus-containing compounds.

2. SOLVENTS

Note: If the detergent Luminox (or equivalent) is used, solvent rinses are not required.

2.1. Use pesticide grade isopropanol as the rinse solvent in routine equipment cleaning procedures. This grade of alcohol must be purchased from a laboratory supply vendor.

2.2. Other solvents, such as acetone or methanol, may be used as the final rinse solvent if they are pesticide grade. However, methanol is more toxic to the environment and acetone may be an analyte of interest for volatile organics.

2.2.1. **Do not use** acetone if volatile organics are of interest.

2.3. Properly dispose of all wastes according to applicable regulations. Containerize all solvents (including rinsates) for on-site remediation or off-site disposal, as required.

2.4. Pre-clean equipment that is heavily contaminated (see FC 1120, section 3) with organic analytes with reagent grade acetone and hexane or other suitable solvents.

2.5. Use pesticide grade methylene chloride when cleaning sample containers.

2.6. Store all solvents away from potential sources of contamination (gas, copier supplies, etc.).

3. ANALYTE-FREE WATER SOURCES

3.1. Analyte-free water is water in which all analytes of interest and all interferences are below method detection limits.

3.2. Maintain documentation (such as results from equipment blanks) to demonstrate the reliability and purity of analyte-free water source(s).

3.3. The source of the water must meet the requirements of the analytical method and must be free from the analytes of interest. In general, the following water types are associated with specific analyte groups:

- Milli-Q (or equivalent polished water): suitable for all analyses.
- Organic-free: suitable for volatile and extractable organics.
- Deionized water: not suitable for volatile and extractable organics if the analytes of interest are present in concentrations that affect the result.
- Distilled water: not suitable for volatile and extractable organics, metals or ultra-trace metals.

3.4. Use analyte-free water for blank preparation and the final decontamination water rinse.

3.5. In order to minimize long-term storage and potential leaching problems, obtain or purchase analyte-free water just prior to the sampling event. If obtained from a source (such as a laboratory), fill the transport containers and use the contents for a single sampling event. Empty the transport container(s) at the end of the sampling event.

3.6. Discard any analyte-free water that is transferred to a dispensing container (such as a wash bottle) at the end of each sampling day.

4. ACIDS

4.1. Reagent Grade Nitric Acid: 10 - 15% (one volume concentrated nitric acid and five volumes deionized water).

4.1.1. Use for the acid rinse unless nitrogen components (e.g., nitrate, nitrite, etc.) are to be sampled.

4.1.2. If sampling for ultra-trace levels of metals, use an ultra-pure grade acid.

4.2. Reagent Grade Hydrochloric Acid: 10% hydrochloric acid (one volume concentrated hydrochloric and three volumes deionized water).

4.2.1. Use when nitrogen components are to be sampled.

4.3. If samples for both metals and the nitrogen-containing components (see FC 1001, section 4.1.1 above) are collected with the equipment, use the hydrochloric acid rinse, or thoroughly rinse with hydrochloric acid after a nitric acid rinse.

4.4. If sampling for ultra trace levels of metals, use an ultra-pure grade acid.

4.5. Freshly prepared acid solutions may be recycled during the sampling event or cleaning process. Dispose appropriately at the end of the sampling event, cleaning process or if acid is discolored or appears otherwise contaminated (e.g., floating particulates).

4.5.1. Transport only the quantity necessary to complete the sampling event.

4.6. Dispose of any unused acids according to FDEP and local ordinances.

FC 1002. *Reagent Storage Containers*

The contents of all containers must be clearly marked.

1. DETERGENTS: Store in the original container or in a high density polyethylene (HDPE) or polypropylene (PP) container.

2. SOLVENTS

2.1. Store solvents to be used for cleaning or decontamination in the original container until use in the field. If transferred to another container for field use, the container must be either glass or Teflon.

2.2. Use dispensing containers constructed of glass, Teflon, or stainless steel. Note: if stainless steel sprayers are used, any components (including gaskets and transfer lines) that contact the solvents must be constructed of inert materials.

3. ANALYTE-FREE WATER: Transport in containers appropriate to the type of water to be stored. If the water is commercially purchased (e.g., grocery store), use the original containers when transporting the water to the field. Containers made of glass, Teflon, polypropylene, or Polyethylene (PE) are acceptable.

3.1. Use glass, Teflon, polypropylene or PE to transport organic-free sources of water on-site.

3.2. Dispense water from containers made of glass, Teflon, PE or polypropylene.

3.3. Do not store water in transport containers for more than three days before beginning a sampling event.

3.4. Store and dispense acids using containers made of glass, Teflon, PE or polypropylene.

FC 1003. *General Requirements*

1. Before using any equipment, clean/decontaminate all sampling equipment (pumps, tubing, lanyards, split spoons, etc.) that are exposed to the sample.

1.1. Before installing, clean (or obtain as certified precleaned) all equipment that is dedicated to a single sampling point and remains in contact with the sample medium (e.g., permanently installed groundwater pump (see FS 2220, section 3.3.4).

1.2. Clean this equipment any time it is removed for maintenance or repair.

1.3. Replace dedicated tubing if discolored or damaged.

2. Clean all equipment in a designated area having a controlled environment (house, laboratory, or base of field operations) and transport to the field precleaned and ready to use, unless otherwise justified.

3. Rinse all equipment with water after use, even if it is to be field-cleaned for other sites. Rinse equipment used at contaminated sites or used to collect in-process (e.g., untreated or partially treated wastewater) samples immediately with water.

4. Whenever possible, transport sufficient clean equipment to the field so that an entire sampling event can be conducted without the need for cleaning equipment in the field.

5. Segregate equipment that is only used once (i.e., not cleaned in the field) from clean equipment and return to the in-house cleaning facility to be cleaned in a controlled environment.

6. Protect decontaminated field equipment (including well sounders) from environmental contamination by securely wrapping and sealing with one of the following:

6.1. Aluminum foil (commercial grade is acceptable);

6.2. Untreated butcher paper; or

6.3. Clean, untreated, disposable plastic bags. Plastic bags may be used:

- For all analyte groups except volatile and extractable organics;
- For volatile and extractable organics, if the equipment is first wrapped in foil or butcher paper or if the equipment is completely dry.

7. Containerize all solvent rinsing wastes, detergent wastes and other chemical wastes requiring off-site or regulated disposal. Dispose of all wastes in conformance with applicable regulations.

FC 1100. Cleaning Sample Collection Equipment

FC 1110. ON-SITE/IN-FIELD CLEANING

1. Cleaning equipment on-site is not recommended because:

1.1. Environmental conditions cannot be controlled.

1.2. Wastes (solvents and acids) must be containerized for proper disposal.

2. If performed, follow the appropriate cleaning procedure as outlined in FC 1130. Ambient temperature water may be substituted in the hot, sudsy water bath, and hot water rinses.

Note: Properly dispose of all solvents and acids.

3. Rinse all equipment with water after use, even if it is to be field-cleaned for other sites. Rinse equipment used at contaminated sites or used to collect in-process (e.g., untreated or partially treated wastewater) samples immediately with water.

FC 1120. HEAVILY CONTAMINATED EQUIPMENT

In order to avoid contaminating other samples, isolate heavily contaminated equipment from other equipment and thoroughly decontaminate the equipment before further use. Equipment is considered heavily contaminated if it:

- Has been used to collect samples from a source known to contain significantly higher levels than background;
- Has been used to collect free product; or
- Has been used to collect industrial products (e.g., pesticides or solvents) or their by-products.

1. Cleaning heavily contaminated equipment in the field is not recommended.

2. ON-SITE PROCEDURES

2.1. Protect all other equipment, personnel and samples from exposure by isolating the equipment immediately after use.

2.2. At a minimum, place the equipment in a tightly sealed untreated plastic bag.

2.3. Do not store or ship the contaminated equipment next to clean, decontaminated equipment, unused sample containers, or filled sample containers.

2.4. Transport the equipment back to the base of operations for thorough decontamination.

2.5. If cleaning must occur in the field, and in order to document the effectiveness of the procedure, collect and analyze blanks on the cleaned equipment (see FQ 1000).

3. CLEANING PROCEDURES

3.1. If organic contamination cannot be readily removed with scrubbing and a detergent solution, prerinse equipment by thoroughly rinsing or soaking the equipment in acetone.

3.1.1. Do not use solvent soaks or rinses if the material is clear acrylic.

3.1.2. Use hexane only if preceded and followed by acetone.

3.2. In extreme cases, it may be necessary to steam clean the field equipment before proceeding with routine cleaning procedures.

3.3. After the solvent rinses (and/or steam cleaning), use the appropriate cleaning procedure (see FC 1130).

3.3.1. Scrub, rather than soak all equipment with sudsy water.

3.3.2. If high levels of metals are suspected and the equipment cannot be cleaned without acid rinsing, soak the equipment in the appropriate acid. Do not use stainless steel equipment when heavy metal contamination is suspected or present, since stainless steel cannot be exposed to prolonged acid soaks.

3.4. If the field equipment cannot be cleaned utilizing these procedures, discard unless further cleaning with stronger solvents and/or oxidizing solutions is effective as evidenced by visual observation and blanks.

3.5. Clearly mark or disable all discarded equipment to discourage use.

FC 1130. GENERAL CLEANING

Follow these procedures when cleaning equipment under controlled conditions. See FC 1110 for modifications if cleaning is performed on-site. Check manufacturer's instructions for cleaning restrictions and/or recommendations.

FC 1131. *Procedure for Teflon, Stainless Steel and Glass Sampling Equipment*

This procedure must be used when sampling for **ALL** analyte groups: extractable organics, metals, nutrients, etc. or if a single decontamination protocol is desired to clean all Teflon, stainless steel and glass equipment.

1. Rinse equipment with hot tap water.

2. Soak equipment in a hot, sudsy water solution (Liqui-Nox or equivalent - see FC 1001, section 1).

3. If necessary, use a brush to remove particulate matter or surface film.

4. Rinse thoroughly with hot tap water.

5. If samples for trace metals or inorganic analytes will be collected with the equipment and the equipment **is not** stainless steel, thoroughly rinse (wet all surfaces) with the appropriate acid solution (see FC 1001, section 4).

6. Rinse thoroughly with analyte-free water. Use enough water to ensure that all equipment surfaces are thoroughly flushed with water.

7. If samples for volatile or extractable organics will be collected, rinse with isopropanol. Wet equipment surfaces thoroughly with free-flowing solvent. Rinse thoroughly with analyte-free water (see FC 1001, section 3).

8. Allow to air dry. Wrap and seal according to FC 1003, section 6 as soon as the equipment is air-dried.

9. If isopropanol is used, the equipment may be air-dried without the final analyte-free water rinse (see FC 1131, section 8 above); however, **the equipment must be completely dry before wrapping or use**.

10. Wrap clean sampling equipment according to the procedure described in FC 1003, section 6.

FC 1132. *General Cleaning Procedure for Plastic Sampling Equipment*

1. Rinse equipment with hot tap water.

2. Soak equipment in a hot, sudsy water solution (Liqui-Nox or equivalent - see FC 1001, section 1).

3. If necessary, use a brush to remove particulate matter or surface film.

4. Rinse thoroughly with hot tap water.

5. Thoroughly rinse (wet all surfaces) with the appropriate acid solution (see FC 1001, section 4). Check manufacturer's instructions for cleaning restrictions and/or recommendations.

6. Rinse thoroughly with analyte-free water. Use enough water to ensure that all equipment surfaces are thoroughly flushed with water. Allow to air dry as long as possible.

7. Wrap clean sampling equipment according to the procedure described in FC 1003, section 6.

FC 1133. *Cleaning Procedure by Analyte Group*

See Table FC 1000-1 for the procedures to be used to decontaminate equipment based on construction of sampling equipment, and analyte groups to be sampled.

FC 1140. AUTOMATIC SAMPLERS, SAMPLING TRAINS AND BOTTLES

1. When automatic samplers are deployed for extended time periods, clean the sampler using the following procedures when routine maintenance is performed. Inspect deployed samplers prior to each use. At a minimum, change the tubing if it has become discolored or has lost elasticity (FC 1140, section 2.3 below).

2. Clean all automatic samplers (such as ISCO) as follows:

   2.1. Wash the exterior and accessible interior portions of the automatic samplers (excluding the waterproof timing mechanisms) with laboratory detergent (see FC 1001, section 1) and rinse with tap water.

2.2. Clean the face of the timing case mechanisms with a clean, damp cloth.

2.3. Check all tubing (sample intake and pump tubing). Change the tubing every six months (if used frequently) or if it has become discolored (i.e., affected by mold and algae) or if it has lost its elasticity.

2.4. See FC 1160, section 4 for the procedures associated with cleaning the tubing in the pump head.

3. AUTOMATIC SAMPLER ROTARY FUNNEL AND DISTRIBUTOR

3.1. Clean with hot sudsy water and a brush (see FC 1001, section 1 for appropriate detergent type).

3.2. Rinse thoroughly with analyte-free water.

3.3. Air dry.

3.4. Replace in sampler.

4. SAMPLER METAL TUBE: Clean as outlined in FC 1160, section 5.

5. REUSABLE GLASS COMPOSITE SAMPLE CONTAINERS

5.1. If containers are used to collect samples that contain oil, grease or other hard to remove materials, it may be necessary to rinse the container several times with reagent-grade acetone before the detergent wash. If material cannot be removed with acetone, discard the container.

5.2. Wash containers following the procedure outlined in FC 1131 above. End with a final solvent rinse if organics are to be sampled.

5.3. Invert containers to drain and air dry for at least 24 hours.

5.4. Cap with aluminum foil, Teflon film or the decontaminated Teflon-lined lid.

5.5. After use, rinse with water in the field, seal with aluminum foil to keep the interior of the container wet, and return to the laboratory or base of operations.

5.6. **Do not recycle or reuse containers if**:

5.6.1. They were used to collect in-process (i.e., untreated or partially treated) wastewater samples at industrial facilities;

5.6.2. A visible film, scale or discoloration remains in the container after the cleaning procedures have been used; or

5.6.3. The containers were used to collect samples at pesticide, herbicide or other chemical manufacturing facilities that produce toxic or noxious compounds. Such containers must be properly disposed of (preferably at the facility) at the conclusion of the sampling activities.

5.6.4. If the containers described above are reused, check no less than 10% of the cleaned containers for the analytes of interest **before** use. If found to be contaminated, (i.e., constituents of interest are found at method detection levels or higher), then **discard the containers**.

6. REUSABLE PLASTIC COMPOSITE SAMPLE CONTAINERS

6.1. Follow FC 1132.

6.2. Inspect the containers. Determine if the containers can be reused by the criteria in FC 1140, section 5 above.

7. GLASS SEQUENTIAL SAMPLE BOTTLES FOR AUTOMATIC SAMPLER BASED FOR SEQUENTIAL MODE

7.1. Clean glass sequential sample bottles to be used for collecting inorganic samples by using a laboratory dishwasher (see FC 1140, sections 7.1.1 through 7.1.3 below) or manually following the procedures in FC 1131.

7.1.1. Rinse with appropriate acid solution (see FC 1001, section 4).

7.1.2. Rinse thoroughly with tap water.

7.1.3. Wash in dishwasher at wash cycle, using laboratory detergent cycle, followed by tap and analyte-free water rinse cycles.

7.2. Replace bottles in covered, automatic sampler base; cover with aluminum foil for storage.

7.3. Rinse bottles in the field with water as soon as possible after sampling event.

8. Glass Sequential Sample Bottles (Automatic Sampler based for Sequential Mode) to be used for Collecting Samples for Organic Compounds

8.1. Use cleaning procedures outlined in FC 1131. Allow containers to thoroughly air dry before use.

8.2. Replace bottles in covered, automatic sampler base; cover with aluminum foil for storage.

9. BOTTLE SIPHONS USED TO TRANSFER SAMPLES FROM COMPOSITE CONTAINERS

9.1. Rinse tubing with solvent and dry overnight in a drying oven.

9.2. Cap ends with aluminum foil and/or Teflon film for storage.

9.3. Seal in plastic for storage and transport.

9.4. Flush siphon thoroughly with sample before use.

10. REUSABLE TEFLON COMPOSITE MIXER RODS

10.1. Follow procedures outlined in FC 1131.

10.2. Wrap in aluminum foil for storage.

FC 1150. FILTRATION EQUIPMENT

1. Dissolved Constituents using in-line, Molded and Disposable Filter Units

1.1. Peristaltic Pump

1.1.1. Clean the pump following procedures in FC 1170, section 2.2.

1.1.2. Clean the pump head tubing following FC 1160, section 4.

1.1.3. If Teflon tubing is used, clean following the procedures in FC 1160, section 3.

1.1.4. Clean other tubing types such as polyethylene according to the appropriate procedures listed in FC 1160, section 7.

1.2. Other Equipment Types (e.g., pressurized Teflon bailer)

1.2.1. Follow the appropriate cleaning regimen specified in FC 1131 through FC 1132 for other types of equipment that utilize in-line, molded and disposable filters.

2. Dissolved Constituents using Non-disposable Filtration Units (e.g., syringes, "tripod assembly")

2.1. Stainless Steel or Glass Units

2.1.1. Follow FC 1131, assembling and applying pressure to the apparatus after each rinse step (water and acid) to drive rinsing solution through the porous filter holder in the bottom of the apparatus.

2.1.2. Remove and clean any transfer tubing according to the appropriate cleaning procedures (see FC 1160).

2.1.3. Assemble the unit and cap both the pressure inlet and sample discharge lines (or whole unit if a syringe) with aluminum foil to prevent contamination during storage.

2.1.4. If the unit will **not** be used to filter volatile or extractable organics, seal the unit in an untreated plastic bag to prevent contamination.

2.2. Reusable In-Line Filter Holders

2.2.1. Clean, using FC 1131, (if Teflon, glass or stainless steel) or FC 1132 (if plastic) assembling and applying pressure to the apparatus after each rinse step (water and acid) to drive rinsing solution through the porous filter holder in the bottom of the apparatus.

2.2.2. Assemble the unit and wrap with aluminum foil to prevent contamination during storage.

2.2.3. If the unit will **not** be used to filter volatile or extractable organics, seal the unit in an untreated plastic bag to prevent contamination.

3. FILTERS

3.1. Do not clean filters. Instructions for rinsing the filters prior to use are discussed in the applicable sampling SOPs (FS 2000 - FS 8000).

FC 1160. SAMPLE TUBING DECONTAMINATION

1. Check tubing:

1.1. For discoloration: Remove discolored tubing from use until it can be cleaned. If the discoloration cannot be removed, discard the tubing.

1.2. For elasticity (if used in a peristaltic-type pump): Discard any tubing that has lost its elasticity.

2. Transport all tubing to the field in precut, **precleaned** sections.

3. TEFLON, POLYETHYLENE AND POLYPROPYLENE TUBING

3.1. New Tubing: Follow this procedure unless the manufacturer/supplier provides certification that the tubing is clean.

3.1.1. Teflon

3.1.1.1. Rinse outside of tubing with pesticide-grade solvent (see FC 1001, section 2).

3.1.1.2. Flush inside of tubing with pesticide-grade solvent.

3.1.1.3. Dry overnight in drying oven or equivalent (zero air, nitrogen, etc.).

3.1.2. Polyethylene and Polypropylene

3.1.2.1. Clean the exterior and interior of the tubing by soaking in hot, sudsy water.

3.1.2.2. Thoroughly rinse the exterior and interior of the tubing with tap water, followed by analyte-free water.

3.2. Reused Tubing

Use the following procedure for in-lab cleaning. **Field cleaning is not recommended**:

3.2.1. Clean the exterior of the tubing by soaking in hot, sudsy water (see FC 1001, section 1) in a stainless steel sink (or equivalent non-contaminating material). Use a brush to remove any particulates, if necessary.

3.2.2. Use a small bottle brush and clean the inside of the tubing ends where the barbs are to be inserted or cut 1-2 inches from the ends of the tubing after cleaning.

3.2.3. Rinse tubing exterior and ends liberally with tap water.

3.2.4. Rinse tubing surfaces and ends with the appropriate acid solution (see FC 1001, section 4), tap water, isopropanol (see FC 1001, section 2), and finally analyte-free water.

3.2.4.1. Note: Eliminate the isopropanol rinse for polyethylene or polypropylene tubing.

3.2.5. Place tubing on fresh aluminum foil or clean polyethylene sheeting. Connect all of the precut lengths of tubing with Teflon inserts or barbs.

3.2.6. Cleaning configuration:

3.2.6.1. Place cleaning reagents: [sudsy water (see FC 1001, section 1); acid (see FC 1001, section 4); isopropanol (see FC 1001, section 2)] in an appropriately cleaned container (2-liter glass jar is recommended).

3.2.6.2. Place one end of the Teflon tubing into the cleaning solution.

3.2.6.3. Attach the other end of the Teflon tubing set to the influent end of a pump.

3.2.6.4. Recycle the effluent from the pump by connecting a length of Teflon tubing from the effluent to the glass jar with the cleaning reagents.

3.2.6.5. Recycling as described above may be done for all reagents listed in FC 1160, section 3.2.6.1 above, **except** the final isopropanol rinse and the final analyte-free water rinse. Disconnect the tubing between the effluent end of the pump and the jar of cleaning reagents.

3.2.6.6. Containerize isopropanol in a waste container for proper disposal.

3.2.6.7. Analyte-free water may be discarded down the drain.

3.2.7. Using the above configuration described in FS 1160, section 3.2.6 above:

3.2.7.1. Pump hot, sudsy water through the connected lengths. Allow the pump to run long enough to pump at least three complete tubing volumes through the tubing set.

3.2.7.2. Using the same procedure, successively pump tap water, the acid solution(s), tap water, isopropanol, and finally analyte-free water through the system.

3.2.7.3. Leave the Teflon inserts or barbs between the precut lengths and cap or connect the remaining ends.

3.2.8. After the interior has been cleaned as described in FC 1160, section 3.2.7 above, rinse the exterior of the tubing with analyte-free water.

3.2.9. Wrap the connected lengths in aluminum foil or untreated butcher paper and store in a clean, dry area until use.

4. Flexible Tubing used in Pump Heads of Automatic Samplers and other Peristaltic Pumps

Replace tubing after each sampling point if samples are collected through the tubing. Unless the pump is deployed to collect samples from the same location over a long period of time, remove and wash the tubing after each sampling event (see FC 1140, section 1).

4.1. Flush tubing with hot tap water then sudsy water (see FC 1001, section 1).

4.2. Rinse thoroughly with hot tap water.

4.3. Rinse thoroughly with analyte-free water.

4.4. If used to collect metals samples, flush the tubing with an appropriate acid solution (see FC 1001, section 4), followed by thorough rinsing with analyte-free water. If used to collect both metals and nitrogen components use hydrochloric acid (see FC 1001, section 4.1.1).

4.5. Install tubing in peristaltic pump or automatic sampler.

4.6. Cap both ends with aluminum foil or equivalent.

Note: Change tubing at specified frequencies as part of routine preventative maintenance.

5. STAINLESS STEEL TUBING

Clean the exterior and interior of stainless steel tubing as follows:

5.1. Using sudsy water (see FC 1001, section 1), scrub the interior and exterior surfaces.

5.2. Rinse with hot tap water.

5.3. Rinse with analyte-free water.

5.4. If volatile or extractable organics are to be sampled, rinse all surfaces with isopropanol (see FC 1001, section 2). Use enough solvent to wet all surfaces with free flowing solvent.

5.5. Allow to air dry or thoroughly rinse with analyte-free water.

6. GLASS TUBING

6.1. Use new glass tubing.

6.2. If volatile or extractable organics are to be sampled, rinse with isopropanol (see FC 1001, section 2).

6.3. Air dry for at least 24 hours.

6.4. Wrap in aluminum foil or untreated butcher paper to prevent contamination during storage.

6.5. Discard tubing after use.

7. MISCELLANEOUS NON-INERT TUBING TYPES (TYGON, RUBBER, PVC, ETC.)

7.1. New Tubing

7.1.1. As a general rule, new tubing may be used without preliminary cleaning.

7.1.2. Protect new tubing from potential environmental contamination by wrapping in aluminum foil and sealing in untreated plastic bags or keep in the original sealed packaging until use.

7.1.3. If new tubing is exposed to potential contamination, rinse the exterior and interior tubing surfaces with hot tap water followed by a thorough rinse with analyte-free water.

7.1.4. If new tubing is to be used to collect samples, thoroughly rinse the tubing with sample water (i.e., pump sample water through the tubing) before collecting samples.

7.2. Reused Tubing

7.2.1. Flush tubing with sudsy solution of hot tap water and laboratory detergent (see FC 1001, section 1).

7.2.2. Rinse exterior and interior thoroughly with hot tap water.

7.2.3. Rinse exterior and interior thoroughly with analyte-free water.

7.2.4. If used to collect only metals samples, flush the tubing with nitric acid (see FC 1001, section 4.1), followed by a thorough rinse with analyte-free water.

7.2.5. If used to collect metals and nitrogen-containing compounds, see FC 1001, section 4.3.

7.2.6. Cap ends in aluminum foil and store in clean, untreated plastic bags to prevent contamination during storage and transport.

FC 1170. PUMPS

1. SUBMERSIBLE PUMPS

1.1. Pumps used for Purging and Sampling Metals and/or Volatile and Extractable Organics

1.1.1. Construction of pump body and internal mechanisms (bladders, impellers, etc.), including seals and connections, must follow Tables FS 1000-1, FS 1000-2 and FS 1000-3.

1.1.2. Tubing material must follow Tables FS 1000-1, FS 1000-2 and FS 1000-3.

1.1.3. Clean pump exterior following FC 1132. Note: omit the solvent rinse if the pump body is constructed of plastic (e.g., ABS, PVC, etc.).

1.1.4. Clean the pump internal cavity and mechanism as follows:

1.1.4.1. If used only for purging, thoroughly flush the pump with water before purging the next well.

1.1.4.2. When used for purging and sampling, completely disassemble the pump (if practical) and decontaminate between each well.

1.1.4.3. When used for purging and sampling and the pump cannot be (practicably) disassembled, then clean the internal cavity/mechanism by pumping

several gallons of sudsy water (see FC 1001, section 1), followed by several gallons of tap water, and finally, several gallons of analyte-free water.

1.1.4.4. If multiple sampling points are located in an area that is not accessible by a vehicle, and it is difficult to return to the vehicle for cleaning or to transport all cleaning materials to the staging location, at a minimum thoroughly rinse the pump with water.

1.1.5. Refer to FC 1160, section 3 to clean Teflon tubing.

1.1.6. Refer to FC 1160, section 5 for stainless steel tubing.

1.1.7. Clean other types of tubing according to FC 1160, sections 6 and 7.

1.2. Pumps used for Purging and Sampling all Analytes except Metals, Volatile and Extractable Organics

1.2.1. Pump construction: no restrictions.

1.2.2. Pump tubing material: no restrictions.

1.2.3. Scrub the exterior of the pump with appropriate metal-free, phosphate-free or ammonia-free detergent solution.

1.2.4. Rinse the exterior with tap water and analyte-free water.

1.2.5. Rinse the interior of the pump and tubing by pumping tap or analyte-free water through the system using a clean bucket or drum.

2. ABOVE-GROUND PUMPS USED FOR PURGING AND SAMPLING

2.1. Pumps used only for Purging

2.1.1. The exterior of the pump must be free of oil and grease.

2.1.2. Select tubing according to Tables FS 1000-1, FS 1000-2 and FS 1000-3.

2.1.3. Clean the tubing that contacts the formation water according to the appropriate protocol for construction materials specified in FC 1160.

2.2. Pumps used for Sampling

2.2.1. Clean the exterior of the pump with a detergent solution followed by a tap water rinse. Use clean cloths or unbleached paper towels that have been moistened with the appropriate solution to wipe down the pump.

2.2.2. Select tubing according to Tables FS 1000-1, FS 1000-2 and FS 1000-3.

2.2.3. Clean the tubing that contacts the formation water according to the appropriate protocol for construction materials specified in FC 1160.

FC 1180. ANALYTE-FREE WATER CONTAINERS

This section pertains to containers that are purchased to transport, store and dispense analyte-free water. It does not apply to water that has been purchased in containers. See FC 1002, section 3 for appropriate construction materials.

1. NEW CONTAINERS

1.1. Wash containers and caps according to FC 1131, omitting the solvent rinse if plastic (polyethylene or polypropylene) containers are being cleaned.

1.2. Cap with Teflon film or the bottle cap. The bottle cap must be composed of the same material as the container and cannot be lined.

2. REUSED CONTAINERS

2.1. Immediately after emptying, cap with aluminum foil, Teflon film or the container cap.

2.2. Wash the exterior of the container with lab-grade detergent solution (see FC 1001, section 1) and rinse with analyte-free water.

2.3. Rinse the interior thoroughly with analyte-free water.

2.4. Invert and allow to drain and dry.

FC 1190. ICE CHESTS AND SHIPPING CONTAINERS

1. Wash the exterior and interior of all ice chests with laboratory detergent (see FC 1001, section 1) after each use.

2. Rinse with tap water and air dry before storing.

3. If the ice chest becomes severely contaminated with concentrated waste or other toxic or hazardous materials clean as thoroughly as possible, render unusable, and properly dispose.

FC 1200. Field Instruments and Drilling Equipment

FC 1210. FIELD INSTRUMENTS (TAPES, METERS, ETC.)

Follow manufacturer's recommendations for cleaning instruments. At a minimum:

1. Wipe down equipment body, probes, and cables with lab-grade detergent solution (see FC 1001, section 1). Check manufacturer's instructions for recommendations and/or restrictions on cleaning.

2. Rinse thoroughly with tap water.

3. Rinse thoroughly with analyte-free water.

4. Store equipment according to the manufacturer's recommendation or wrap equipment in aluminum foil, untreated butcher paper or untreated plastic bags to eliminate potential environmental contamination.

FC 1220. SOIL BORING EQUIPMENT

This section pertains only to equipment that is not used to collect samples. Clean split spoons, bucket augers and other sampling devices according to FC 1131.

1. Remove oil, grease, and hydraulic fluid from the exterior of the engine and power head, auger stems, bits and other associated equipment with a power washer or steam jenny or wash by hand with a brush and sudsy waster (no degreasers).

2. Rinse thoroughly with tap water.

FC 1230. WELL CASING CLEANING

These are recommended procedures for cleaning well casing and riser pipes. Use procedures specified by a FDEP contract, order, permit, or rule, if different or more stringent than the procedures outlined below.

1. FDEP recommends only using casing that is designed for subsurface environmental groundwater monitoring.

2. Casing that has been contaminated with grease, hydraulic fluid, petroleum fuel, etc. may require additional cleaning or deemed unusable.

3. All casings and riser pipes should be cleaned before installation, unless the casing is received wrapped and ready for installation:

3.1. Steam clean all casings and riser pipes except PVC. Steam cleaning criteria shall meet the following: water pressure - 2500 psi; water temperature - 200°F.

3.2. Rinse thoroughly with tap (potable) water. This tap water must be free of the analytes of interest.

FC 1300. Sample Containers

FC 1310. Obtaining Clean Containers

1. Obtain clean sample containers in one of three ways:

1.1. From commercial vendors as precleaned containers. The cleaning grades must meet EPA analyte specific requirements. Keep all records for these containers (lot numbers, certification statements, date of receipt, etc.) and document the container's intended uses;

1.2. From internal groups within the organization that are responsible for cleaning and maintaining containers according to the procedures outlined in FC 1320; or

1.3. From a subcontracted laboratory that is accredited under the National Environmental Laboratory Accreditation Program (NELAP).

1.3.1. The contractor must verify that the laboratory follows the container cleaning procedures outlined in FC 1320.

1.3.2. If the laboratory cleaning procedures are different, the contractor must require that the laboratory use the following cleaning procedures or provide documentation and historical records to show that their in-house procedure produces containers that are free from the analytes of interest.

FC 1320. Container Cleaning Procedures

1. Refer to Table FC 1000-2. Follow the cleaning steps in the order specified in the chart.

2. Cleaning procedures that are different from those outlined in FC 1320 may be used as long as blanks collected in the containers are free from the analytes of interest and any analytical interferences and the cleaning procedures are supported by historical and continuing documentation.

3. Inspect all containers before cleaning.

3.1. **Do not recycle or reuse containers if**:

3.1.1. Containers were used to collect in-process (i.e., untreated or partially treated) wastewater samples at industrial facilities;

3.1.2. A visible film, scale or discoloration remains in the container after the cleaning procedures have been used; or

3.1.3. Containers were used to collect samples at pesticide, herbicide or other chemical manufacturing facilities that produce toxic or noxious compounds. Such containers shall be properly disposed of (preferably at the facility) at the conclusion of the sampling activities.

3.1.4. If the containers described above are reused, check no less than 10% of the cleaned containers for the analytes of interest before use. If found to be contaminated (i.e., analytes of interest are found at MDL levels or higher), discard the containers.

FC 1400. Documentation

Document cleaning procedures described below for the indicated activities. See FD 1000 for additional information about required records and retention of documents.

FC 1410. FIELD EQUIPMENT

1. IN-FIELD CLEANING

   1.1. Initially identify the procedures that are used to clean equipment in the field by SOP numbers and dates of usage.

   1.2. Record the date and time that equipment was cleaned.

2. IN-HOUSE CLEANING

   2.1. Retain any cleaning certificates, whether from a laboratory or commercial vendor.

   2.2. Identify the procedure(s) that are used to clean equipment by the SOP number and dates of usage.

   2.3. Record the date that the equipment was cleaned.

FC 1420. SAMPLE CONTAINERS

1. Organizations that order precleaned containers must retain the packing slips, and lot numbers of each shipment, any certification statements provided by the vendor and the vendor cleaning procedures.

2. Organizations that clean containers must maintain permanent records of the following:

   2.1. Procedure(s) used to clean containers by SOP number and dates of usage.

   2.2. If containers are certified clean by the laboratory the laboratory must record:

   - Type of container;
   - Date cleaned;
   - SOP used;
   - Person responsible for cleaning;
   - Lot number (date of cleaning may be used) of the batch of containers that were cleaned using the same reagent lots and the same procedure;
   - The results of quality control tests that were run on lot numbers; and
   - Any additional cleaning or problems that were encountered with a specific lot.

FC 1430. REAGENTS AND OTHER CLEANING SUPPLIES

Maintain a record of the lot number with the inclusive dates of use for all acids, solvents, and other cleaning supplies.

Appendix FC 1000
Tables, Figures and Forms

Table FC 1000-1
Procedures for Decontamination at the Base of Operations or On-Site

Construction Material	Analyte Group Sampled	SOP Reference	Base of Operations	On-Site
Teflon or Glass	All	FC 1131	Follow as written	May substitute ambient temperature water for the hot water rinses and hot detergent solution
	Extractable & Volatile Organics Petroleum Hydrocarbons		May omit acid rinse	May substitute ambient temperature water for the hot water rinses and hot detergent solution May omit acid rinse
	Metals[l] Radionuclides For ultra trace metals, refer to FS 8200		May omit solvent rinse	May substitute ambient temperature water for the hot water rinses and hot detergent solution May omit solvent rinse
	Inorganic Nonmetallics Physical & Aggregate Properties Aggregate Organics Biologicals Volatile Inorganics		May omit solvent rinse	Rinse several times with water Rinse several times with sample water from the next sampling location
	Microbiological – Viruses Microbiological - Bacteria		Omit solvent and acid rinses	Rinse several times with water Rinse several times with sample water from the next sampling location
Metallic (stainless steel, brass, etc.)	All Extractable & Volatile Organics Petroleum Hydrocarbons	FC 1131	Omit the acid rinse	May substitute ambient temperature water for the hot water rinses and hot detergent solution Omit the acid rinse
	Metals Radionuclides		Omit the acid rinse May omit the solvent rinse	May substitute ambient temperature water for the hot water rinses and hot detergent solution Omit the acid rinse May omit the solvent rinse
	Inorganic Nonmetallics Physical & Aggregate Properties Aggregate Organics Biologicals Volatile Inorganics		Omit solvent rinse May omit the acid rinse	Rinse several times with water Rinse several times with sample water from the next sampling location

Table FC 1000-1
Procedures for Decontamination at the Base of Operations or On-Site

Construction Material	Analyte Group Sampled	SOP Reference	Base of Operations	On-Site
	Microbiological – Viruses Microbiological - Bacteria		Omit solvent and acid rinses	Rinse several times with water Rinse several times with sample water from the next sampling location
Plastic (Polyethylene, polypropylene, PVC, silicone, acrylic	Volatile and Extractable Organics;	FC 1132	Follow as written.	May substitute ambient temperature water for the hot water rinses and hot detergent solution
	Inorganic Nonmetallics Physical & Aggregate Properties Aggregate Organics Biologicals Volatile Inorganics		May omit the acid rinse	Rinse several times with water Rinse several times with sample water from the next sampling location
	Microbiological – Viruses Microbiological - Bacteria		Omit acid rinse	Rinse several times with water Rinse several times with sample water from the next sampling location

[i] Do not use glass if collecting samples for boron or silica.

Table FC 1000-2
Container Cleaning Procedures

ANALYSIS / ANALYTE GROUP	CLEANING STEPS See Description Below
Extractable Organics	1, 2, 4, 6 (not required if Luminox (or equivalent is used), (5 and 7 optional), 11
Volatile Organics	1, 2, 4, (6 optional, methanol only), 7
Metals	1, 2, 3, 4, 8, 11 ** **Procedures to clean containers for ultra-trace metals are found in FS 8200
Inorganic Nonmetallics, Radionuclides, Physical and Aggregate Properties, Aggregate Inorganics, and Volatile Inorganics	1, 2, 3*, 4, 8, 11 * For nutrients, replace nitric acid with hydrochloric acid, or use a hydrochloric acid rinse after the nitric acid rinse. See FC 1001, section 4
Petroleum Hydrocarbons, and Oil and Grease	1, 2, 3, 4, (5, 6, 7 optional), 11
Microbiological (all)	1, 2, 4, 8, 9, 11
Toxicity Tests (Includes Bioassays)	1, 2, 10, 2, 4, 6.1, (10 optional), 11

NOTE: Steps 1 and 2 may be omitted when cleaning new, uncertified containers.

1. Wash with hot tap water and a brush using a suitable laboratory-grade detergent:

   1.1. Volatile and Extractable Organics, Petroleum Hydrocarbon, Oil and Grease: Luminox, Liqui-Nox, Alconox or equivalent;

   1.2. Inorganic nonmetallics: Liqui-Nox or equivalent;

   1.3. Metals: Liqui-Nox, Acationox, Micro or equivalents:

   1.4. Microbiologicals (all): Must pass an inhibitory residue test.

2. Rinse thoroughly with hot tap water.
3. Rinse with 10% nitric acid solution.
4. Rinse thoroughly with analyte-free water (deionized or better).
5. Rinse thoroughly with pesticide-grade methylene chloride.
6. Rinse thoroughly with pesticide-grade isopropanol, acetone or methanol.

   6.1. For bioassays, use only acetone, and only when containers are glass.

7. Oven dry at 103°C to 125°C for at least 1 hour.

Table FC 1000-2
Container Cleaning Procedures

7.1. VOC vials and containers must remain in the oven in a contaminant-free environment until needed. They should be capped in a contaminant-free environment just prior to dispatch to the field.

8. Invert and air-dry in a contaminant-free environment.

9. Sterilize containers:

9.1. Plastic: 60 min at 170°C, loosen caps to prevent distortion.

9.2. Glass: 15 min at 121°C.

10. Rinse with 10% hydrochloric acid followed by a sodium bicarbonate solution.

11. Cap tightly and store in a contaminant-free environment until use. Do not use glass if collecting samples for boron or silica.

FS 2200. Groundwater Sampling

1. INTRODUCTION AND SCOPE

1.1 Use these Standard Operating Procedures to collect groundwater samples. They are designed to ensure that the collected samples will be representative of water in the aquifer or target formation and that the samples have not been altered or contaminated by the sampling and handling procedures. These procedures apply to permanently and temporarily installed monitoring wells, wells constructed using "direct-push" techniques, wells with installed plumbing, remedial groundwater treatment systems and excavations where groundwater is present. Use of alternative, DEP-approved and properly documented procedures (e.g., Corporate SOP, ASTM Standards, alternative equipment, etc.) is acceptable if they meet the intent (e.g., sample representativeness and integrity) of this standard (see FA 1000).

1.2 The topics in this SOP include equipment and supply selection, equipment construction materials, and purging and sampling techniques.

1.3 Use the following DEP SOPs in conjunction with FS 2200:

- FA 1000 Regulatory Scope and Administrative Procedures for Use of DEP SOPs
- FC 1000 Cleaning/Decontamination Procedures
- FD 1000 Documentation Procedures
- FQ 1000 Field Quality Control Requirements
- FS 1000 General Sampling Procedures
- FS 2000 General Aqueous Sampling
- FT 1000 Field Testing and Measurement
- FT 1100 Field pH
- FT 1200 Field Specific Conductance
- FT 1400 Field Temperature
- FT 1500 Field Dissolved Oxygen
- FT 1600 Field Turbidity

2. Groundwater samples may be collected from a number of different configurations. Each configuration is associated with a unique set of sampling equipment requirements and techniques:

3. Wells without Plumbing: These wells require that equipment be brought to the well to purge and sample unless dedicated equipment is placed in the well.

4. Wells with In-Place Plumbing: Wells with in-place plumbing do not require that equipment be brought to the well to purge and sample. In-place plumbing is generally considered permanent equipment routinely used for purposes other than purging and sampling, such as for water supply. They are generally found at wellfields, industrial facilities, and private residences. See FS 2300 for procedures to sample potable water wells. Air Strippers or Remedial Systems: These types of systems are installed as remediation devices. Sample these wells like drinking water wells (see FS 2300).

FS 2201 *Equipment and Supplies*

Use groundwater purging and sampling equipment constructed of only non-reactive, non-leachable materials that are compatible with the environment and the selected analytes. In selecting groundwater purging and sampling equipment, give consideration to the depth of the well, the depth to groundwater, the volume of water to be evacuated, the sampling and purging technique, and the analytes of interest. Refer to Tables FS 1000-1, FS 1000-2, FS 1000-3 and FS 2200-1 for selection of appropriate equipment.

Additional supplies such as reagents, preservatives, and field measurement equipment are often necessary.

1. FLOW CONTAINER: DEP recommends using a flow-through cell or container when collecting measurements for purging stabilization. The design must ensure that fresh formation water continuously contacts the measuring devices and does not aerate the sample or otherwise affect the groundwater properties.

2. PUMPS: All pumps or pump tubing must be lowered and retrieved from the well slowly and carefully to minimize disturbance to the formation water. This is especially critical at the air/water interface. Avoid the resuspension of sediment particles (turbidity) at the bottom of the well or adhered to the well casing during positioning of the pump or tubing.

2.1 Above-Ground Pumps

2.1.1 Variable Speed Peristaltic Pump: Use a variable speed peristaltic pump to purge groundwater from wells when the static water level in the well is no greater than 20-25 feet below land surface (BLS). If the water levels are deeper than 18-20 feet BLS, the pumping velocity will decrease.

2.1.1.1 A variable speed peristaltic pump can be used for normal purging and sampling (see FS 2213 and FS 2221), sampling low permeability aquifers or formations (see FS 2222) and collecting filtered groundwater samples (see FS 2225, section 1).

2.1.1.2 Most analyte groups can be sampled with a peristaltic pump if the tubing and pump configurations are appropriate. See Table FS 1000-3 for proper tubing selection and pump configurations.

2.1.2 Variable Speed Centrifugal Pump: A variable speed centrifugal pump can be used to purge groundwater from 2-inch and larger internal diameter wells. Do not use this type of pump to collect groundwater samples.

2.1.2.1 When purging is complete, do not allow the water that remains in the tubing to fall back into the well. Install a check valve at the end of the purge tubing, and withdraw the tubing slowly from the well while the pump is still running.

2.1.2.2 See Table FS 1000-3 for proper tubing selection and allowable analyte groups.

2.2 Submersible Pumps

2.2.1 Variable Speed Electric Submersible Pump: A variable speed submersible pump can be used to purge and sample groundwater from 2-inch and larger internal diameter wells.

2.2.1.1 A variable speed submersible pump can be used for normal purging and sampling (see FS 2213 and FS 2221), sampling low permeability aquifers or

formations (see FS 2222) and collecting filtered groundwater samples (see FS 2225, section 1).

2.2.1.2 Make sure that the pump housing, fittings, check valves and associated hardware are constructed of stainless steel. Make sure that any other materials are compatible with the analytes of interest. See Table FS 1000-3 for restrictions.

2.2.1.3 Install a check valve at the output side of the pump to prevent backflow.

2.2.1.4 If purging and sampling for organics:

- The entire length of the delivery tube must be Teflon, Polyethylene or Polypropylene (PP) tubing.
- The electrical cord must be sealed in Teflon, Polyethylene or PP and any cabling must be sealed in Teflon, Polyethylene or PP, or be constructed of stainless steel.
- All interior components that contact the sample water (impeller, seals, gaskets, etc.) must be constructed of stainless steel or Teflon.

2.2.2 Variable Speed Bladder Pump: A variable speed positive displacement bladder pump (no-gas contact) can be used to purge and sample groundwater from 3/4-inch and larger internal diameter wells.

2.2.2.1 A variable speed bladder pump can be used for normal purging and sampling (see FS 2213 and FS 2221), sampling low permeability aquifers or formations (see FS 2222) and collecting filtered groundwater samples (see FS 2225, section 1).

2.2.2.2 The bladder pump system is composed of the pump, the compressed air tubing, the water discharge tubing, the controller and a compressor or compressed gas supply.

2.2.2.3 The pump consists of a bladder and an exterior casing or pump body that surrounds the bladder and two (2) check valves. These parts can be composed of various materials, usually combinations of polyvinyl chloride (PVC), Teflon, Polyethylene, PP and stainless steel. Other materials must be compatible with the analytes of interest. See Table FS 1000-3 for restrictions.

2.2.2.4 If purging and sampling for organics:

- The pump body must be constructed of stainless steel and the valves and bladder must be Teflon, Polyethylene or PP̃
- The entire length of the delivery tube must be Teflon, Polyethylene or PP.
- Any cabling must be sealed in Teflon̩ Polyethylene or PP, or be constructed of stainless steel.
- Permanently installed pumps may have a PVC pump body as long as the pump remains in contact with the water in the well.

3. BAILERS:

3.1 Purging: DEP does not recommend using bailers for purging unless no other equipment can be used or purging with a bailer has been specifically authorized by a DEP program, permit, contract or order (see Table FS 2200-3). Use a bailer if there is non-aqueous phase liquid (free product) in the well or non-aqueous phase liquid is suspected to

be in the well. If in doubt about the appropriateness of using a bailer at a site or during a particular sampling event, contact the appropriate DEP program or project manager. If a bailer is used, follow FS 2213, section 4, with no deviations.

3.2 Sampling: Bailers may be used to routinely collect some analyte groups or under specific circumstances for other analyte groups (see Table FS 2200-3).

3.3 Construction and Type:

3.3.1 Bailers must be constructed of materials compatible with the analytes of interest. See Table FS 1000-3 for restrictions.

3.3.2 Stainless steel, Teflon, Polyethylene and PP bailers may be used to sample all analytes.

3.3.3 Use disposable bailers when sampling grossly contaminated sample sources.

3.3.4 DEP recommends using dual check valve bailers when collecting samples.

3.3.5 Use bailers with a controlled flow bottom when collecting volatile organic samples.

3.3.6 Use bailers that can be pressurized when collecting filtered samples for metals.

3.4 Contamination Prevention:

3.4.1 Keep the bailer wrapped (foil, butcher paper, etc.) until just before use.

3.4.2 Use protective gloves to handle the bailer once it is removed from its wrapping.

3.4.3 Handle the bailer by the lanyard to minimize contact with the bailer surface.

4. LANYARDS

4.1 Lanyards must be made of non-reactive, non-leachable material such as cotton twine, nylon, or stainless steel; or, coated with Teflon, Polyethylene or PP.

4.1.1 Evaluate the appropriateness of the lanyard material with analyses of equipment blanks for the analytes of interest, as necessary.

4.2 Discard cotton twine, nylon, and non-stainless steel braided lanyards after sampling each monitoring well.

4.3 Decontaminate stainless steel, coated Teflon, Polyethylene and PP lanyards between monitoring wells (see FC 1003). They do not need to be decontaminated between purging and sampling operations.

4.4 Securely fasten lanyards to downhole equipment (bailers, pumps, etc.).

4.5 Do not allow lanyards used for downhole equipment to touch the ground surface.

FS 2210. GROUNDWATER PURGING

Perform procedures in the following sections to calculate purging parameters and to purge groundwater from monitoring wells, wells with installed plumbing, high-volume wells, air stripper systems and other remedial treatment systems.

FS 2211 *Water Level and Purge Volume Determination*

Collect representative groundwater samples from the aquifer. The amount of water that must be purged from a well is determined by the volume of water and/or field parameter stabilization.

1. GENERAL EQUIPMENT CONSIDERATIONS

1.1 Selection of appropriate purging equipment depends on the analytes of interest, the well diameter, transmissivity of the aquifer, the depth to groundwater and other site conditions.

1.2 Use a pump to purge the well.

1.3 Use a bailer if there is non-aqueous phase liquid in the well or non-aqueous phase liquid is suspected to be in the well.

1.4 Bailers may be used if approved by a DEP program, or if bailer use is specified in a permit, contract or DEP order (see Table FS 2200-3). If used, bailers must be of appropriate type and construction, and the user must follow the procedure outlined in FS 2213, section 4, with no deviations. If in doubt about the appropriateness of using a bailer at a site or during a particular sampling event, contact the appropriate DEP program or project manager. DEP does not recommend using bailers because improper bailing:

1.4.1 Introduces atmospheric oxygen which precipitates metals (i.e., iron) or causes other changes in the chemistry of the water in the sample (i.e., pH)

1.4.2 Agitates groundwater which biases volatile and semi-volatile organic analyses due to volatilization

1.4.3 Agitates the water in the aquifer and resuspends fine particulate matter

1.4.4 Surges the well, loosening particulate matter in the annular space around the well screen

1.4.5 Introduces dirt into the water column if the sides of the casing wall are scraped

2. INITIAL INSPECTION

2.1 Verify the identification of the monitoring well by examining markings, sign plates, placards or other designations.

2.2 Remove the well cover and remove all standing water around the top of the well casing (manhole) before opening the well cap.

2.3 Inspect the exterior protective casing of the monitoring well for damage and document the results of the inspection if there is a problem.

2.4 It is recommended that you place a protective covering around the well head. Replace the covering if it becomes soiled or ripped.

2.5 Inspect the well lock and determine whether the cap fits tightly. Replace the cap if necessary.

3. WATER LEVEL MEASUREMENTS: Use an electronic probe or chalked tape to determine the water level.

3.1 General Procedures

Perform these steps using either the electronic probe or chalked tape method.

3.1.1 Decontaminate all equipment that will contact the groundwater in the well before use.

3.1.2 Measure the depth to groundwater from the top of well casing to the nearest 0.01 foot and always measure from the same reference point or survey mark on the well casing. If there is no reference mark, measure from the north side of the casing.

3.1.3 Record the measurement and the reference point.

3.2 Electronic Probe

3.2.1 Follow the manufacturer's instructions for use.

3.2.2 Record the measurement.

3.3 Chalked Line Method: This method is not recommended if collecting samples for organic or inorganic parameters.

3.3.1 Lower chalked tape into the well until the lower end is in the water (usually determined by the sound of the weight hitting the water).

3.3.2 Record the length of the tape relative to the reference point (see section 3.2 above).

3.3.3 Quickly remove the tape from the well.

3.3.4 Record the length of the wetted portion to the nearest 0.01 foot.

3.3.5 Determine the depth to water by subtracting the length of the wetted portion (see section 3.5.3 above) from the total length (see section 3.5.2 above). Record the result.

4. WATER COLUMN DETERMINATION

4.1 Do not determine the total depth of the well by lowering the probe to the bottom of the well immediately before purging and sampling. If the well must be sounded, delay purging and sampling activities for at least 24 hours after the well was sounded or for a time sufficient to meet the purge stabilization criterion for turbidity. Alternatively, collect samples before sounding the well.

4.2 Subtract the depth to the top of the water column from the total well depth to determine the length of the water column.

4.3 The total well depth depends on the well construction. Some wells may be drilled in areas of sinkhole or karst formations or rock leaving an open borehole. Attempt to find the total borehole depth in cases where there is an open borehole below the cased portion.

5. WELL WATER VOLUME

5.1 Calculate the total volume of water in gallons in the well using the following equation:

V = (0.041)d x d x h

Where: V = volume in gallons

d = well diameter in inches

h = height of the water column in feet

5.2 The total volume of water in the well may also be determined with the following equation by using a casing volume per foot factor (Gallons per Foot of Water) for the appropriate diameter well:

V = [Gallons per Foot of Water] x h

Where: V = volume in gallons

h = height of the water column in feet

Casing Internal Diameter	Approximate Gallons per Foot of Water
0.75”	0.02
1”	0.04
1.25”	0.06
2”	0.16
3”	0.37
4”	0.65
5”	1.02
6”	1.47
12”	5.88

5.3 Record all measurements and calculations in the field records.

6. Purging Equipment Volume

Calculate the total volume of the pump, associated tubing and container that is used for in situ measurements (flow container), if used, using the following equation:

V = p + ((0.041)d x d x l) + fc

Where: V = volume in gallons

p = volume of pump in gallons

d = tubing diameter in inches

l = length of tubing in feet

fc = volume of flow cell in gallons

7. When collecting samples from multiple wells on a site, if the groundwater elevation data are to be used to construct groundwater elevation contour maps, all water level measurements must be taken within the same 24-hour time interval unless a shorter time period is required by a DEP program. If the site is tidally influenced, complete the water level measurements within the time frame of an incoming or outgoing tide.

FS 2212 *Well Purging Techniques*

The selection of the purging technique and equipment is dependent on the hydrogeologic properties of the aquifer, especially depth to groundwater and hydraulic conductivity. The intent of proper purging is to stabilize the water level in the well and minimize the hydraulic stress to the hydrogeologic formation.

Every attempt must be made to match the pumping rate with the recharge rate of the well before evaluating the purging completion criteria.

A flowchart which summarizes purging procedure options is presented in Figure FS 2200-2.

Select equipment using the construction and configuration requirements specified in Table FS 2200-1. See the discussions in FS 2201.

1. MEASURING THE PURGE VOLUME: The volume of water that is removed during purging must be recorded. Measure the volume during the purging operation.

1.1 Collect the water in a graduated container and multiply the number of times the container was emptied by the volume of the container, or

1.2 Estimate the volume based on pumping rate. Use this technique only if the pumping rate is constant. Determine the pumping rate by measuring the amount of water that is pumped for a fixed period of time or use a flow meter.

1.2.1 Calculate the amount of water that is discharged per minute:

$$D = \frac{\text{Measured amount}}{\text{Total time in minutes}}$$

1.2.2 Calculate the time needed to purge one (1) well volume or one (1) purging equipment volume:

$$\text{Time} = \frac{V}{D}$$

Where: V = well volume determined from FS 2211, section 5, or purging equipment volume

D = discharge rate calculated in section 1.2.1. above

1.2.3 Make new measurements (see section 1.2.1 above) each time the pumping rate is changed, or

1.3 Use a totalizing flow meter.

1.3.1 Record the reading on the totalizer prior to purging.

1.3.2 Record the reading on the totalizer at the end of purging.

1.3.3 Subtract the reading on the totalizer prior to purging from the reading on the totalizer at the end of purging to obtain the volume purged.

1.4 Record in the field records the times that purging begins and ends.

2. Stabilization Measurement Frequency

2.1 Begin to record stabilization measurements after pumping the minimum volume as prescribed in options 2.3 – 2.5 below. Every attempt must be made to match the pumping rate with the recharge rate of the well before evaluating the purging criteria.

2.2 If the well screened interval is not known, use option 2.3, below.

2.3 Wells with Fully Submerged Screen and Pump or Intake Tubing Placed at the Top of the Water Column (conventional purge): Purge until the water level has stabilized (well recovery rate equals the purge rate), then purge a minimum of one (1) well volume prior to collecting measurements of the stabilization parameters. Allow at least one quarter (1/4) well volume to purge between subsequent measurements.

2.4 Wells with Fully Submerged Screen and Pump or Intake Tubing Placed Within the Screened Interval (minimizing purge volume): Purge until the water level has stabilized (well recovery rate equals the purge rate), then purge a minimum of one (1) volume of the pump, associated tubing and flow container (if used) prior to collecting measurements of the stabilization parameters. Take measurements of the stabilization parameters no sooner

than two (2) minutes apart. Purge at least three (3) volumes of the pump, associated tubing and flow container, if used, prior to collecting a sample.

If the water level drops into the screened interval during purging, lower the pump or tubing intake as in FS 2213, section 1.3 below and follow purging procedures for partially submerged well screens (2.5 below).

2.5 Wells with a Partially Submerged Well Screen: Purge until the water level has stabilized (well recovery rate equals the purge rate), then purge a minimum of one (1) well volume prior to collecting measurements of the stabilization parameters. Take measurements of the stabilization parameters no sooner than two (2) minutes apart.

3. PURGING COMPLETION: DEP recommends the use of a flow-through container to measure the stabilization parameters discussed below. Alternatively, measure all parameters *in situ* by inserting measurement probes into the well at the depth appropriate for the purging option. Purging is considered complete if the criteria in section 3.1, 3.2 or 3.3 below are satisfied. Make every attempt to satisfy the criteria in section 3.1. Every attempt must be made to match the pumping rate with the recharge rate of the well before evaluating the purging criteria.

3.1 Three (3) consecutive measurements of the five (5) parameters listed below must be within the stated limits. The measurements evaluated must be the last three consecutive measurements taken before purging is stopped. The range between the highest and the lowest values for the last three measurements of temperature, pH and specific conductance cannot exceed the stated limits. The last three consecutive measurements of dissolved oxygen and turbidity must all be at or below the listed thresholds.

- Temperature: ± 0.2° C
- pH: ± 0.2 Standard Units
- Specific Conductance: ± 5.0% of reading
- Dissolved Oxygen: ≤20% Saturation
- Turbidity: ≤20 NTU

3.2 Naturally occurring conditions may prevent attaining the ≤20% saturation criterion for dissolved oxygen, typically in surficial aquifers. See section 3.5, below.

3.3 Naturally occurring conditions may prevent attaining the ≤20 NTU criterion for turbidity. However, when collecting groundwater samples for metals or certain inorganic (e.g., phosphorus forms) or extractable organic (e.g. polynuclear aromatic hydrocarbons) chemicals, make every attempt to reduce turbidity to ≤20 NTU to avoid a potential turbidity-associated bias for these analytes. See section 3.5, below.

3.4 Document and report the following, as applicable, except that the last four (4) items only need to be submitted once:

- Purging rate.
- Drawdown in the well, if any.
- Pump or tubing intake placement.
- Length and location of the screened interval.
- A description of the process and the data used to design the well.
- The equipment and procedure used to install the well.

- The well development procedure.
- Pertinent lithologic or hydrogeologic information.

3.5 If the criteria in section 3.1 above for dissolved oxygen and/or turbidity cannot be met, then three (3) consecutive measurements of the five (5) parameters listed below must be within the stated limits.

3.5.1 The measurements evaluated must be the last three consecutive measurements taken before purging is stopped. The range between the highest and the lowest values for the last three measurements cannot exceed the stated limits.

- Temperature: ± 0.2° C
- pH: ± 0.2 Standard Units
- Specific Conductance: ± 5.0% of reading
- Dissolved Oxygen: ± 0.2 mg/L or 10%, whichever is greater
- Turbidity: ± 5 NTUs or 10%, whichever is greater

3.5.2 Additionally, document and report the following, as applicable, except that the last four (4) items only need to be submitted once:

- Purging rate.
- Drawdown in the well, if any.
- Pump or tubing intake placement.
- Length and location of the screened interval.
- A description of conditions at the site that cause the dissolved oxygen to be high and/or dissolved oxygen measurements made within the screened or open borehole portion of the well with a downhole dissolved oxygen probe.
- A description of conditions at the site that cause the turbidity to be high and any procedures that will be used to minimize turbidity in the future.
- A description of the process and the data used to design the well.
- The equipment and procedure used to install the well.
- The well development procedure.
- Pertinent lithologic or hydrogeologic information.

3.5.3 If from review of the submitted data the Department determines that both the elevated Dissolved Oxygen and Turbidity measurements are due to naturally occurring conditions, then only the first four (4) items are required to be submitted in future reports. However, if the Department cannot determine if the Dissolved Oxygen or Turbidity is elevated due to naturally occurring conditions, then in addition to the first four (4) items, a description of the conditions at the site that caused the affected parameter(s) to be high is required to be submitted in future reports.

3.6 If the stabilization parameters in either section 3.1 or 3.2 cannot be met, and all attempts have been made to minimize the drawdown, check the instrument condition and calibration, purging flow rate and all tubing connections to determine if they might be affecting the ability to achieve stable measurements. All measurements that were made during the attempt must be documented. The sampling team leader may decide whether or

not to collect a sample or to continue purging after five (5) well volumes (conventional purge section 2.1 or 2.3 above) or five (5) volumes of the screened interval (minimizing purge volumes in section 2.2 above).

Further, the report in which the data are submitted must include the following, as applicable, except that the last four (4) items only need to be submitted once:

- Purging rate.
- Pump or tubing intake placement.
- Length and location of the screened interval.
- Drawdown in the well, if any.
- A description of conditions at the site that caused the dissolved oxygen to be high and/or dissolved oxygen measurements made within the screened or open borehole portion of the well with a downhole dissolved oxygen probe.
- A description of conditions at the site that caused the turbidity to be high and any procedures that will be used to minimize turbidity in the future.
- A description of the process and the data used to design the well.
- The equipment and procedure used to install the well.
- The well development procedure.
- Pertinent lithologic or hydrogeologic information.

If from review of the submitted data the DEP determines that both the elevated Dissolved Oxygen and Turbidity measurements are due to naturally occurring conditions, then only the first four (4) items are required to be submitted in future reports. However, if the DEP cannot determine if the Dissolved Oxygen or Turbidity is elevated due to naturally occurring conditions, then in addition to the first four (4) items, a description of the conditions at the site that caused the affected parameter(s) to be high is required to be submitted in future reports.

3.7 One fully dry purge (not recommended). This criterion applies only if purging was attempted per FS 2212, FS 2213, and section 3.4.1 below, and if it is impossible to balance the pumping rate with the rate of recharge at very low pumping rates (< 100 mL/minute).

3.7.1 If wells have previously and consistently purged dry, when purged according to FS 2212 and FS 2213, and the current depth to groundwater indicates that the well will purge dry during the current sampling event, minimize the amount of water removed from the well by using the same pump to purge and collect the sample:

3.7.1.1 Place the pump or tubing intake within the well screened interval.

3.7.1.2 Use very small diameter Teflon, Polyethylene or PP tubing and the smallest possible pump chamber volume to minimize the total volume of water pumped from the well and to reduce drawdown.

3.7.1.3 Select tubing that is thick enough to minimize oxygen transfer through the tubing walls while pumping.

3.7.1.4 Pump at the lowest possible rate (100 mL/minute or less) to reduce drawdown to a minimum.

3.7.1.5 Purge at least two (2) volumes of the pumping system (pump, tubing and flow cell, if used).

3.7.1.6 Measure pH, Specific Conductance, Temperature, Dissolved Oxygen and Turbidity and begin to collect the samples (see FS 2222).

4. Collect samples immediately after purging is complete.

4.1 The time period between completing the purge and sampling cannot exceed six (6) hours.

4.2 If sample collection does not occur within one (1) hour of purging completion, re-measure the five (5) field parameters Temperature, pH, Specific Conductance, Dissolved Oxygen and Turbidity just prior to collecting the sample.

4.2.1 If the measured values are not within 10 percent of the previous measurements, re-purge the well.

4.2.2 See section 3.4 above when collecting samples from wells that have purged dry.

FS 2213 *Purging Wells Without Plumbing (Monitoring Wells)*

1. TUBING/PUMP PLACEMENT

1.1 Do not lower the pump or intake hose (tubing) to the bottom of the well. Pump or tubing placement procedures will be determined by the purging option selected in FS 2212, section 2 above or FS 2214 below.

1.1.1 Minimizing Purge Volume: If the following conditions can be met, position the intake hose (tubing) or pump in the screened or open borehole interval.

- The same pump must be used for both purging and sampling,
- The well screen or borehole interval must be less than or equal to 10 feet, and
- The well screen or borehole must be fully submerged.

1.1.2 If the position or length of the screened interval or open borehole is unknown or estimated, place the intake hose (tubing) or pump to perform conventional purging in 1.2 below.

1.1.3 Position the pump or intake hose when purging large-diameter deep wells with open boreholes using the procedure in FS 2214 below.

1.2 Conventional Purging: Position the pump or intake tubing in the top one foot of the water column or no deeper than necessary for the type of pump.

1.2.1 If purging with a bailer, see section 4 below.

1.3 Partially Submerged Screened Interval: If the well screen or open borehole is partially submerged, and the pump will be used for both purging and sampling, position the pump or intake hose (tubing) in the portion of the water column within the submerged screened or open borehole interval.

1.3.1 If the position or length of the screened interval or open borehole is unknown or estimated, place the intake hose (tubing) or pump to perform conventional purging in 1.2 above.

1.3.2 Purge large-volume, high-recharge wells as in FS 2214 below.

1.3.3 If purging with a bailer, see section 4 below.

2. NON-DEDICATED (PORTABLE) PUMPS

2.1 Variable Speed Peristaltic Pump

2.1.1 Install a new, 1-foot maximum length of silicone tubing in the peristaltic pump head.

2.1.2 Attach a short section of tubing to the discharge side of the pump-head silicone tubing and into a graduated container.

2.1.3 Attach one end of a length of new or precleaned transport tubing to the intake side of the pump head silicone tubing.

2.1.4 Place the transport tubing in the monitoring well per one of the options in FS 2213, section 1 above.

2.1.5 Measure the depth to groundwater at frequent intervals.

2.1.6 Record these measurements.

2.1.7 Adjust the purging rate so that it is equivalent to the well recovery rate to minimize drawdown.

2.1.8 If the purging rate exceeds the well recovery rate, reduce the pumping rate to balance the withdrawal rate with the recharge rate.

2.1.9 If the water table continues to drop during pumping, lower the tubing at the approximate rate of drawdown so that the water is removed from the top of the water column.

2.1.10 Record the purging rate each time the rate changes.

2.1.11 Measure the purge volume by one of the methods outlined in FS 2212, section 1.

2.1.12 Record this measurement.

2.1.13 Decontaminate the pump and tubing between wells (see FC 1000) or only the pump if precleaned tubing is used for each well.

2.2 Variable Speed Centrifugal Pump

2.2.1 Position fuel powered equipment **downwind** and at least 10 feet from the well head. Make sure that the exhaust faces downwind.

2.2.2 Place the decontaminated suction hose so that water is always pumped from the top of the water column.

2.2.3 Equip the suction hose with a foot valve to prevent purge water from re-entering the well.

2.2.4 Measure the depth to groundwater at frequent intervals.

2.2.5 Record these measurements.

2.2.6 Adjust the purging rate so that it is equivalent to the well recovery rate to minimize drawdown.

2.2.7 If the purging rate exceeds the well recovery rate, reduce the pumping rate to balance the withdrawal rate with the recharge rate.

2.2.8 If the water table continues to drop during pumping, lower the tubing at the approximate rate of drawdown so that the water is removed from the top of the water column.

2.2.9 Record the purging rate each time the rate changes.

2.2.10 Measure the purge volume by one of the methods outlined in FS 2212, section 1.

2.2.11 Record this measurement.

2.2.12 Decontaminate the pump and tubing between wells (see FC 1000) or only the pump if precleaned tubing is used for each well.

2.3 Variable Speed Electric Submersible Pump

2.3.1 Position fuel powered equipment downwind and at least 10 feet from the well head. Make sure that the exhaust faces downwind.

2.3.2 Carefully position the decontaminated pump per one of the options in FS 2213, section 1 above.

2.3.3 Measure the depth to groundwater at frequent intervals.

2.3.4 Record these measurements.

2.3.5 Adjust the purging rate so that it is equivalent to the well recovery rate to minimize drawdown.

2.3.6 If the purging rate exceeds the well recovery rate, reduce the pumping rate to balance the withdrawal rate with the recharge rate.

2.3.7 If the water table continues to drop during pumping, lower the tubing or pump at the approximate rate of drawdown so that the water is removed from the top of the water column.

2.3.8 Record the purging rate each time the rate changes.

2.3.9 Measure the purge volume by one of the methods outlined in FS 2212, section 1.

2.3.10 Record this measurement.

2.3.11 Decontaminate the pump and tubing between wells (see FC 1000) or only the pump if precleaned tubing is used for each well.

2.4 Variable Speed Bladder Pump

2.4.1 Position fuel powered equipment **downwind** and at least 10 feet from the well head. Make sure that the exhaust faces downwind.

2.4.2 Attach the tubing and carefully position the pump per one of the options in FS 2213, section 1 above.

2.4.3 Measure the depth to groundwater at frequent intervals.

2.4.4 Record these measurements.

2.4.5 Adjust the purging rate so that it is equivalent to the well recovery rate to minimize drawdown.

2.4.6 If the purging rate exceeds the well recovery rate, reduce the pumping rate to balance the withdrawal rate with the recharge rate.

2.4.7 If the water table continues to drop during pumping, lower the tubing or pump at the approximate rate of drawdown so that the water is removed from the top of the water column.

2.4.8 Record the purging rate each time the rate changes.

2.4.9 Measure the purge volume by one of the methods outlined in FS 2212, section 1.

2.4.10 Record this measurement.

2.4.11 Decontaminate the pump and tubing between wells (see FC 1000) or only the pump if precleaned tubing is used for each well.

3. DEDICATED PORTABLE PUMPS: Place dedicated pumps per one of the options in FS 2213, section 1 above.

3.1 Variable Speed Electric Submersible Pump

3.1.1 Position fuel powered equipment **downwind** and at least 10 feet from the well head. Make sure that the exhaust faces downwind.

3.1.2 Measure the depth to groundwater at frequent intervals.

3.1.3 Record these measurements.

3.1.4 Adjust the purging rate so that it is equivalent to the well recovery rate to minimize drawdown.

3.1.5 If the purging rate exceeds the well recovery rate, reduce the pumping rate to balance the withdrawal with the recharge rate.

3.1.6 Record the purging rate each time the rate changes.

3.1.7 Measure the purge volume by one of the methods outlined in FS 2212, section 1.

3.1.8 Record this measurement.

3.2 Variable Speed Bladder Pump

3.2.1 Position fuel powered equipment **downwind** and at least 10 feet from the well head. Make sure that the exhaust faces downwind.

3.2.2 Measure the depth to groundwater at frequent intervals.

3.2.3 Record these measurements.

3.2.4 Adjust the purging rate so that it is equivalent to the well recovery rate to minimize drawdown.

3.2.5 If the purging rate exceeds the well recovery rate, reduce the pumping rate to balance the withdrawal with the recharge rate.

3.2.6 Record the purging rate each time the rate changes.

3.2.7 Measure the purge volume by one of the methods outlined in FS 2212, section 1.

3.2.8 Record this measurement.

4. BAILERS: DEP recommends against using bailers for purging except as a last contingency, or if free product is present in the well or suspected to be in the well. However, they may be used if approved by a DEP program, or specified in a permit, contract or DEP order (see Table FS 2200-3 and FS 2211, section 1.3). If in doubt about the appropriateness of using a bailer at a site or during a particular sampling event, contact the appropriate DEP program or project manager.

4.1 Minimize handling the bailer as much as possible.

4.1.1 Remove the bailer from its protective wrapping just before use.

4.1.2 Attach a lanyard of appropriate material (see FS 2201, section 4).

4.1.3 Use the lanyard to move and position the bailer.

4.2 Lower and retrieve the bailer slowly and smoothly.

4.3 Lower the bailer carefully into the well to a depth approximately a foot above the water column.

4.3.1 Do not lower the top of the bailer more than one (1) foot below the top of the water table so that water is removed from the top of the water column. Ensure that the length of the bailer does not exceed the length of the water column.

4.3.2 Allow time for the bailer to fill with aquifer water as it descends into the water column.

4.4 Carefully raise the bailer.

4.4.1 Retrieve the bailer at the same rate of 2 cm/sec until the bottom of the bailer has cleared to top of the water column.

4.5 Measure the purge volume by one of the methods outlined in FS 2212, section 1.

4.5.1 Record the volume of the bailer.

4.6 Continue to carefully lower and retrieve the bailer as described above until the purging completion conditions specified in FS 2212, section 3, have been satisfied.

4.6.1 Remove at least one (1) well volume before collecting measurements of the field parameters. Take each subsequent set of measurements after removing at least one quarter (1/4) well volume between measurements.

FS 2214 *Purging Large-Volume, High-Recharge Wells With Portable Pumps*

If a well originally constructed for high-flow-rate pumping will be sampled as a monitoring well, use these guidelines to develop a purging procedure applicable to the specific details of the well construction. Typical wells constructed for this purpose may be deep, large-diameter wells with a section of open borehole. Evaluate each well on a case-by-case basis and consider any available information on the construction and hydraulic performance of the well.

1. PURGING PROCEDURE

1.1 Place the pump at the top of the open borehole segment of the well.

1.2 Start purging while monitoring stabilization parameters as in FS 2212, section 3 above.

1.3 Purge at least one equipment volume before measuring stabilization parameters.

1.4 If the well is being purged for the first time using these guidelines, monitor stabilization parameters for an extended period until confident that sufficient volume has been pumped from the open borehole to draw fresh formation water into the pump tubing and flow-through container. Use the information obtained from the first-time purging of the well to determine the pumping rate and duration of purging required for future sampling events at the well.

1.5 Purge at least three equipment volumes before evaluating purging completion.

2. PURGING COMPLETION

2.1 Complete the purging of the well when the last three consecutive measurements of the purge stabilization parameters have met the applicable criteria specified in FS 2212, section 3 above.

3. Collect samples from the well using the procedures in FS 2221, section 1 below.

FS 2215. *Purging Wells With Plumbing (production wells or permanently installed pumps equipped with sampling ports or sampling spigots)*

Wells with in-place plumbing are commonly found at municipal water treatment plants, industrial water supplies, private residences, etc. Depending on the sampling objective for collecting samples using installed plumbing, purge the system and collect samples closest to the point of consumption, or, as close to the source well as possible. When purging is required and the purge volume of the plumbing system is not known, purge the system until the purging completion criteria in FS 2212, section 3, have been met.

1. CONTINUOUSLY RUNNING PUMPS

1.1 Select the spigot that is closest to the pump and before any storage tanks (if possible).

1.2 Remove all hoses, aerators and filters (if possible).

1.3 Open the spigot and purge at maximum flow.

1.4 If a storage tank is located between the pump and the spigot, purge the volume of the tank, lines and spigot.

1.5 If the spigot is before any storage tank, purge until sufficient volume is removed to flush the stagnant water from the spigot and the tap line to the spigot.

1.6 Reduce the flow rate to $\leq$ 500 mL/minute (a 1/8" stream) or approximately 0.1 gal/minute before collecting samples. When sampling for volatile organic compounds, reduce the flow to $\leq$100 mL/minute before collecting the samples.

2. INTERMITTENTLY RUNNING PUMPS

2.1 Select the spigot that is closest to the pump and before any storage tanks (if possible).

2.2 Remove all hoses, aerators and filters (if possible).

2.3 Open the spigot and purge sufficient volume at a maximum, practical flow rate to flush the spigot and lines and until the purging completion criteria in FS 2212, section 3, have been met.

2.4 If a storage tank is located between the pump and the spigot, purge the volume of the tank, lines and spigot.

2.5 Ensure that the purge stabilization measurement of dissolved oxygen is not biased with aeration of the sample by a high flow rate in the flow-through container.

2.6 Reduce the flow rate to $\leq$ 500 mL/minute (a 1/8" stream) or approximately 0.1 gal/minute before collecting samples. When sampling for volatile organic compounds, reduce the flow to $\leq$ 100 mL/minute before collecting the samples.

FS 2216. *Purging Airstrippers and Remedial Treatment Systems*

If collecting samples for groundwater contamination monitoring, follow FS 2215above.

FS 2220. GROUNDWATER SAMPLING TECHNIQUES

1. Purge wells using the techniques outlined in FS 2210.

2. Replace the protective covering around the well if it is soiled or torn after completing the purging operations.

3. EQUIPMENT CONSIDERATIONS

Follow all notes and restrictions as indicated in Table FS 2200-1 and as discussed in FS 2201.

NOTE: The only pumps that are currently approved for use in collecting volatile organic samples through the pump are stainless steel and Teflon variable speed submersible pumps, stainless steel and Teflon or Polyethylene variable speed bladder pumps, and permanently installed PVC bodied pumps (variable speed bladder or submersible pumps) as long as the pump remains in contact with the water in the well at all times.

3.1 Collect the sample into the sample container from the sampling device. **Do not** use intermediate containers.

3.2 In order to avoid contaminating the sample or loss of analytes from the sample:

3.3 Handle the sampling equipment as little as possible.

3.3.1 Minimize the equipment that is exposed to the sample.

3.3.2 Minimize aeration of samples collected for VOC analysis.

3.3.3 Reduce sampling pump flow rates to ≤ 100 mL/minute when collecting VOC samples.

3.4 Dedicated Sampling Equipment

3.4.1 Whenever possible, use dedicated equipment because it significantly reduces the chance of cross-contamination.

3.4.2 Dedicated is defined as equipment that is to be used solely for one location for the life of that equipment (e.g., permanently mounted pump).

3.4.3 All material construction and restrictions from Table FS 2200-1 also apply to dedicated equipment. Purchase equipment with the most sensitive analyte of interest in mind.

3.5 Cleaning/Decontamination

3.5.1 Clean or ensure dedicated pumps are clean before installation. They do not need to be cleaned prior to each use but must be cleaned if they are withdrawn for repair or servicing.

3.5.2 Clean or make sure any permanently mounted tubing is clean before installation.

3.5.3 Change or clean tubing when the pump is withdrawn for servicing.

3.5.4 Clean any replaceable or temporary parts as specified in FC 1000.

3.5.5 Collect equipment blanks on dedicated pumping systems when the tubing is cleaned or replaced.

3.5.6 Clean or ensure dedicated bailers are clean before placing them into the well.

3.5.7 Collect an equipment blank on dedicated bailers before introducing them into the water column.

3.5.8 Suspend dedicated bailers above the water column if they are stored in the well.

FS 2221. *Sampling Wells Without Plumbing*

1. SAMPLING WITH PUMPS: Variable speed stainless steel and Teflon submersible pumps and stainless steel, Teflon or Polyethylene bladder pumps, and permanently installed PVC-bodied variable speed submersible or bladder pumps, as long as the pump remains in contact with the water in the well at all times, may be used to sample for all organics. The delivery tubing must be Teflon, Polyethylene or PP. **Extractable organics** may be collected through a peristaltic pump if ≤ 1 foot of silicone tubing is used in the pump head or a vacuum trap is used (see Figure FS 2200-1 for specific configuration). Follow all notes and restrictions as defined in Table FS 2200-1 and discussed in Equipment and Supplies (FS 2201) when using pumps to collect samples.

Do not lower the pump or tubing to the bottom of the well.

1.1 Peristaltic Pump

1.1.1 Volatile Organics Using Manual Fill and Drain Method: Collect volatile organics last. If the pump tubing is placed within the screened interval, the tubing cannot be reinserted into the well, and steps 1.1.1.3 through 1.1.1.6 below are prohibited.

1.1.1.1 Ensure that there is sufficient tubing volume to fill the requisite number of VOC vials.

1.1.1.2 Remove the drop tubing from the inlet side of the pump.

1.1.1.3 Submerse the drop tubing into the water column and allow it fill.

1.1.1.4 Remove the drop tubing from the well.

1.1.1.5 Prevent the water in the tubing from flowing back into the well.

1.1.1.6 Carefully allow the groundwater to drain by gravity into the sample vials. Avoid turbulence. Do not aerate the sample. The flow rate must be ≤ 100 mL/minute.

1.1.1.7 Repeat steps 1.1.1.3 - 1.1.1.6 until enough vials are filled.

1.1.2 Volatile Organics Using the Pump to Fill and Drain the Tubing: Collect volatile organics last. If the pump tubing is placed within the screened interval, the tubing cannot be reinserted into the well, and steps 1.1.2.2 through 1.1.2.8 below are prohibited.

1.1.2.1 Ensure that there is sufficient tubing volume to fill the requisite number of VOC vials.

1.1.2.2 Submerse the drop tubing into the water column.

1.1.2.3 Use the pump to fill the drop tubing.

1.1.2.4 Quickly remove the tubing from the pump.

1.1.2.5 Prevent the water in the tubing from flowing back into the well.

1.1.2.6 Remove the drop tubing from the well and fill the vials using the pump or gravity-drain methods in steps 1.1.2.7 or 1.1.2.8 below.

1.1.2.7 Reverse the flow on the peristaltic pump to deliver the sample into the vials at a slow, steady rate. The flow rate must be ≤ 100 mL/minute.

1.1.2.8 Or, remove the drop tubing from the inlet side of the pump and carefully allow the groundwater to drain into the sample vials. Avoid turbulence. Do not aerate the sample. The flow rate must be ≤ 100 mL/minute.

1.1.2.9 Repeat steps 1.1.2.2 through 1.1.2.8 until enough vials are filled.

1.1.3 Extractable Organics Collected Through Silicone Pump-Head Tubing:

1.1.3.1 Ensure that a 1-foot maximum length of new silicone tubing was installed in the peristaltic pump head assembly before the well was purged if the same pump is being used to purge and sample the well. Otherwise, install a new length of tubing as described above.

1.1.3.2 Collect extractable organic samples directly from the effluent delivery tubing (attached to discharge side of the silicone pump head tubing) into the sample container.

1.1.3.3 If there is a concern that sample analytes are absorbed, adsorbed, leached or otherwise affected or lost by pumping through the silicone pump-head tubing, sample the well using the organic trap assembly in 1.1.4 below.

1.1.4 Extractable Organics Using an Optional Organic Trap Assembly

1.1.4.1 Assemble the components of the pump and trap according to Figure FS 2200-1.

1.1.4.2 The sample container should be the trap bottle.

1.1.4.3 All equipment that contacts the groundwater **before** the sample container must be constructed of Teflon, Polyethylene, PP, stainless steel or glass, including the transport tubing to and from the sample container, the interior liner of the container cap and all fittings. **Do not use a rubber stopper as a cap.**

1.1.4.4 Connect the outflow tubing from the container to the influent side of the peristaltic pump.

1.1.4.5 Prevent the water in the down-hole delivery tubing from flowing back into the well while performing this connection.

1.1.4.6 Turn the pump on and reduce the flow rate to a smooth and even flow.

1.1.4.7 Discard a small portion of the sample to allow an air space.

1.1.4.8 Preserve (if required), label and complete the field notes.

1.1.5 Inorganics

1.1.5.1 Inorganic samples may be collected from the effluent tubing.

1.1.5.2 If samples are collected from the pump, decontaminate all tubing (including the tubing in the head) or change it between wells.

1.1.5.3 Preserve (if required), label and complete field notes.

1.2 Variable Speed Bladder Pump

1.2.1 If sampling for organics the pump body must be constructed of stainless steel and the valves and bladder must be Teflon. All tubing must be Teflon, Polyethylene, or PP and any cabling must be sealed in Teflon, Polyethylene or PP, or made of stainless steel.

1.2.2 After purging to a smooth even flow, reduce the flow rate.

1.2.3 When sampling for volatile organic compounds, reduce the flow rate to 100 mL/minute or less, if possible.

1.3 Variable Speed Submersible Pump

1.3.1 The housing must be stainless steel.

1.3.2 If sampling for organics, the internal impellers, seals and gaskets must be constructed of stainless steel, Teflon, Polyethylene or PP. The delivery tubing must be Teflon, Polyethylene or PP and the electrical cord must be sealed in Teflon and any cabling must be sealed in Teflon or constructed of stainless steel.

1.3.3 After purging to a smooth even flow, reduce the flow rate.

1.3.4 When sampling for volatile organic compounds, reduce the flow rate to 100 mL/minute or less, if possible.

2. SAMPLING WITH BAILERS: A high degree of skill and coordination are necessary to collect representative samples with a bailer. When properly used, bailers may be used to collect samples for certain analyte groups and under specific conditions (see Table FS 2200-3). They must be of an appropriate type and construction (see FS 2201, section 3), and must be used as outlined below. If in doubt about the appropriateness of using a bailer at a site or during a particular sampling event, contact the appropriate DEP program or project manager.

2.1 General Considerations

2.1.1 Minimize handling the bailer as much as possible.

2.1.1.1 Wear sampling gloves.

2.1.1.2 Remove the bailer from its protective wrapping just before use.

2.1.1.3 Attach a lanyard of appropriate material (see FS 2201, section 4).

2.1.1.4 Use the lanyard to move and position the bailers.

2.1.2 Do not allow the bailer or lanyard to touch the ground.

2.1.3 Rinsing

2.1.3.1 If the bailer is certified precleaned, no rinsing is necessary.

2.1.3.2 If both a pump and a bailer are to be used to collect samples, rinse the exterior and interior of the bailer with sample water from the pump before removing the pump.

2.1.3.3 If the purge pump is not appropriate for collecting samples (e.g., non-inert components), rinse the bailer with by collecting a single bailer of the groundwater to be sampled. Use the technique described in section 2.2, Bailing Technique, below.

2.1.3.4 Discard the water appropriately.

2.1.3.5 **Do not** rinse the bailer if Oil & Grease, TRPHs, etc., (see FS 2006) are to be collected.

2.2 Bailing Technique

2.2.1 Collect all samples that are required to be collected with a pump before collecting samples with the bailer.

2.2.2 Raise and lower the bailer gently to minimize stirring up particulate matter in the well and the water column which can increase sample turbidity.

2.2.3 Lower the bailer carefully into the well to a depth approximately a foot above the water column. Ensure that the length of the bailer does not exceed the length of the water column.

2.2.3.1 When the bailer is in position, lower the bailer into the water column at a rate of 2 cm/sec until the desired depth is reached (see section 2.2.3 above).

2.2.4 Do not lower the top of the bailer more than one (1) foot below the top of the water table so that water is removed from the top of the water column.

2.2.5 Allow time for the bailer to fill with aquifer water as it descends into the water column.

2.2.6 Do not allow the bailer to touch the bottom of the well or particulate matter will be incorporated into the sample.

2.2.6.1 Carefully raise the bailer (see section 2.2.2 above). Retrieve the bailer at the same rate of 2 cm/sec until the bottom of the bailer has cleared to top of the water column.

2.2.7 Lower the bailer to approximately the same depth each time.

2.2.8 Collect the sample.

2.2.8.1 Install a device to control the flow from the bottom of the bailer and discard the first few inches of water. Reduce the flow to $\leq$ 100 mL/minute when collecting VOC samples.

2.2.8.2 Fill the appropriate sample containers by allowing the sample to slowly flow down the side of the container. Minimize aeration of VOC samples.

2.2.8.3 Discard the last few inches of water in the bailer.

2.2.9 Repeat steps 2.2.1 through 2.2.8.3 for additional samples.

2.2.10 Measure the DO, pH, temperature, turbidity and specific conductance after the final sample has been collected.

2.2.10.1 Record all measurements and note the time that sampling was completed.

3. SAMPLING WELLS WITH FLOATING NON-AQUEOUS PHASE LIQUID: DEP does not recommend the sampling of wells with floating non-aqueous phase liquid for trace contaminants. This concerns primarily petroleum related sites, but includes any chemical product (e.g., solvent) that floats on the water table. Sampling is acceptable if the information is to be used for the purpose of remedial design.

Sample data from such wells cannot provide useful information regarding the level of contamination. Furthermore, these wells typically do not provide legitimate data because of permanent chemical contamination from product contact with the well casing for an extended period of time.

DEP does reserve the right to require sampling of these wells, not for levels of trace contaminants, but for confirmation of an appropriate remediation technique. This type of sampling is performed **below** the non-aqueous phase layer (see section 3.2 below).

3.1 Non-Aqueous Phase Liquid Sampling: Non-aqueous phase liquid may be evident in a cased monitoring well or in an open excavation.

3.1.1 Non-aqueous phase liquid is normally sampled for two reasons:

- Documentation for its existence and thickness; and
- Determination of the type of product so that the proper analyses can be performed to determine extent. This is only feasible for relatively recent releases as it may not be possible to identify weathered product.

3.1.2 Disposable plastic (acrylic, clear PVC) bailers are recommended for sampling. Disposable Polyethylene and PP bailers are also acceptable. Other wide mouth vessels may be used for sampling non-aqueous phase liquid in an excavation.

3.1.3 Monitoring Well

3.1.3.1 If a non-aqueous phase liquid is identified in a monitoring well during the water level measurement, measure its thickness in the well. If the thickness of the non-aqueous phase liquid is greater than 0.01 foot or product globules are present, collect a sample using a precleaned disposable bailer.

3.1.3.2 Measure the product thickness to the nearest 0.01 foot after withdrawing the bailer.

3.1.3.3 Pour a portion of the product into a glass sample container.

3.1.3.4 This sample is considered a concentrated waste. Therefore, package the container in protective wrapping to prevent breakage, isolate from other samples, and ice to 4°C.

3.1.4 Excavation

3.1.4.1 If non-aqueous phase liquid is observed in an open excavation, a glass sample container or a precleaned intermediate vessel may be used to collect the sample.

3.1.4.2 Securely tie a lanyard to the container and lower it into the excavation.

3.1.4.3 Gently lower and retrieve the container so that no solid material is released or collected.

3.1.4.4 If sufficient water is available, a bailer can be used.

3.1.4.5 Although not recommended, screened casing can be placed (or augered and placed) in the bottom of the excavation and the product sampled with a bailer.

3.1.4.6 Avoid dangerous situations, such as standing too close to the edge of an excavation, riding in the backhoe bucket, or entering a trench or excavation that may collapse.

3.1.4.7 Follow all applicable OSHA regulations.

3.2 Sampling Below Product

3.2.1 This type of depth-specific sampling to attempt to sample the dissolved constituents in the water column below the product layer is performed only at the request of DEP or its designee.

3.2.2 These data provide information that helps define adequate groundwater treatment. Without these data, incorrect (and sometimes unnecessarily expensive) remediation techniques may be designed for a situation where they are not required.

3.2.3 There are some substantial logistical problems involved with sending a sampler through non-aqueous phase liquid to sample the groundwater below. Although there are some products designed specifically for this type of sampling, they are expensive and the results may not be commensurate with their cost. The use of "self-engineered" equipment or coverings may be the best option.

3.2.4 These data are only to be used for qualitative use and will aid in deciding on an appropriate remediation technique.

3.2.5 Wrapping bailers and tubing in plastic seems to be the most popular technique in getting past the product layer.

3.2.6 Although not recommended, some have wrapped submersible pumps in several layers of plastic and retrieved each layer by a separate lanyard. One suggestion would be to use a rigid piece of stainless steel tubing wrapped in plastic.

3.2.6.1 Once the covered tubing is past the layer, pull up on the plastic, piercing the plastic and exposing the (somewhat) clean tubing inlet.

3.2.6.2 Introduce the wrapped hose slowly to not entrain any more product into the dissolved layer located below.

3.2.6.3 Also, perform this procedure with a peristaltic pump or a vacuum pump linked to a trap bottle. To use this setup, the water table must be no deeper than 15-20 feet, realizing that actual sampling may be occurring several feet below the product layer.

FS 2222. *Sampling Low Permeability Aquifers or Wells That Have Purged Dry*

1. Collect the sample(s) after the well has been purged according to FS 2212, section 3.4. Minimize the amount of water removed from the well by using the same pump to purge and collect the sample. If the well has purged dry, collect samples as soon as sufficient sample water is available.

2. Measure the five (5) field parameters Temperature, pH, Specific Conductance, Dissolved Oxygen and Turbidity at the time of sample collection.

3. Advise the analytical laboratory and the client that the usual amount of sample for analysis may not be available.

FS 2223. *Sampling Wells With In-Place Plumbing*

1. If a storage tank is present, locate a cold water spigot, valve or other sampling point close to the well head between the pump and the storage tank. If there is no sampling location between the pump and the storage tank, locate the spigot, valve or other sampling point closest to the tank.

1.1 Depending on the sampling objective for collecting samples using installed plumbing, purge the system and collect samples closest to the point of consumption, or, as close to the source well as possible.

2. Remove all screens or aerators and reduce the flow rate to no more than 500 mL/minute. If collecting samples for volatile organic compounds, reduce the flow rate to 100 mL/minute or less. Collect the samples directly into the appropriate containers.

FS 2224. *Sampling Airstripper and Remedial Treatment System Sampling*

1. Reduce the flow rate to less than 500 mL/minute and begin sample collection.

2. If collecting samples for volatile organic compounds, reduce the flow rate to 100 mL/minute or less.

3. Collect the samples directly into the appropriate containers.

FS 2225. *Filtering Groundwater Samples*

Filtered groundwater samples can only be collected after approval from the DEP program or project manager. If filtering is approved, the DEP program or permit condition may require both filtered and unfiltered samples to be collected, analyzed and reported.

1. FILTERING GROUNDWATER FOR METALS:

1.1 Unless specified otherwise by the DEP program, use a new, disposable, high capacity, 1-µm in-line filter.

1.2 Use a variable speed peristaltic, bladder or submersible pump with the in-line filter fitted on the outlet end.

1.2.1 Peristaltic pumps, bladder pumps or submersible pumps can be used when water levels are no greater than 20 to 25 feet deep.

1.2.2 Bladder pumps or submersible pumps must be used when water levels are greater than 20 to 25 feet deep.

1.3 Ensure that a 1-foot maximum length of new, silicone tubing was installed in the peristaltic pump head assembly before the well was purged if the same pump is being used to purge and sample the well. Otherwise, install a new length of tubing as described above.

1.4 Ensure that new or precleaned delivery tubing was assembled with the peristaltic pump before the well was purged if the same pump is being used to purge and sample the well. Otherwise, assemble the pump with new or precleaned delivery tubing and the new filter.

1.5 Insert the filter on the high pressure side (i.e., on the delivery side) of the pump.

1.5.1 Flush the filter before attaching to the pump tubing assembly with 30-50 mL of analyte free water or an inert gas (nitrogen) to remove atmospheric oxygen;

1.5.2 Or, with the filter attached to the pump tubing assembly, hold the filter upright with the inlet and outlet in the vertical position and pump water from the aquifer through the filter until all atmospheric oxygen has been removed.

1.6 Collect the filtered samples directly into the sample container from the high-pressure (delivery) side of the pump tubing assembly.

1.6.1 Collect filtered samples by either of the methods in 1.6.1.3 or 1.6.1.4 below if the static water level in the well is too deep for a variable speed peristaltic pump and a variable speed electric submersible pump or variable speed bladder pump is not available.

1.6.1.1 Do not agitate the sample or expose it to atmospheric oxygen.

1.6.1.2 **Do not** pour the sample into any intermediate vessel for subsequent filtration.

1.6.1.3 Collect the sample in a Polyethylene, Teflon or PP bailer that can be pressurized. When the bailer has been retrieved, immediately connect the filter and begin to pressurize the bailer;

1.6.1.4 Or, collect the sample with a bailer and immediately place the intake tube of the peristaltic pump into the full bailer and begin pumping the water through the filter as described in section 1.2 above.

1.7 **Do not** use the following equipment for filtering groundwater samples for metals:

1.7.1 Any pump and apparatus combination in which the filter is on the vacuum (suction) side of the pump.

1.7.2 Any type of syringe or barrel filtration apparatus.

1.7.3 Any filter that is not encased in a one-piece, molded unit.

2. Filtering groundwater for non-metallic analytes

2.1 The following analytes cannot be filtered:

- Oil and Grease
- Total Recoverable Petroleum Hydrocarbons (TRPH)
- FL-PRO
- Volatile Organic Compounds (VOC)
- Microbiological Analytes
- Volatile Inorganic Compounds (e.g., Hydrogen Sulfide)

2.2 Unless specified otherwise by the regulatory program, use a new, disposable, high capacity, 0.45 µm in-line filter.

2.3 Assemble the pump, tubing and filter as in 1.2 – 1.5 above.

2.4 Flush the filter as in 1.5.1 or 1.5.2 above.

2.5 Collect the samples as in 1.6 – 1.6.1.4 above.

Appendix FS 2200

Tables, Figures and Forms

Table FS 2200-1
Equipment for Collecting Groundwater Samples

Activity	Equipment Type
Well Purging	Variable speed centrifugal pump Variable speed submersible pump Variable speed bladder pump Variable speed peristaltic pump Bailer with lanyard: Not Recommended
Well Stabilization	pH meter DO meter Conductivity meter Thermometer/Thermistor Turbidimeter Flow-through cell Multi-function meters
Sample Collection	Variable speed peristaltic pump Variable speed submersible pump Variable speed bladder pump Bailer with lanyard (See Table FS 2200-3)
Filtration	Variable speed peristaltic pump Variable speed submersible pump Variable speed bladder pump Pressurized bailer 1.0 µm high capacity molded filter 0.45 µm high capacity molded filter
Groundwater Level	Electronic sensor Chalked tape

Table FS 2200-2
Dissolved Oxygen Saturation

TEMP	D.O.	mg/L	TEMP	D.O.	mg/L	TEMP	D.O.	mg/L	TEMP	D.O.	mg/L
deg C	SAT.	20%	deg C	SAT.	20%	deg C	SAT.	20%	deg C	SAT.	20%
15.0	10.084	2.017	19.0	9.276	1.855	23.0	8.578	1.716	27.0	7.968	1.594
15.1	10.062	2.012	19.1	9.258	1.852	23.1	8.562	1.712	27.1	7.954	1.591
15.2	10.040	2.008	19.2	9.239	1.848	23.2	8.546	1.709	27.2	7.940	1.588
15.3	10.019	2.004	19.3	9.220	1.844	23.3	8.530	1.706	27.3	7.926	1.585
15.4	9.997	1.999	19.4	9.202	1.840	23.4	8.514	1.703	27.4	7.912	1.582
15.5	9.976	1.995	19.5	9.184	1.837	23.5	8.498	1.700	27.5	7.898	1.580
15.6	9.955	1.991	19.6	9.165	1.833	23.6	8.482	1.696	27.6	7.884	1.577
15.7	9.934	1.987	19.7	9.147	1.829	23.7	8.466	1.693	27.7	7.870	1.574
15.8	9.912	1.982	19.8	9.129	1.826	23.8	8.450	1.690	27.8	7.856	1.571
15.9	9.891	1.978	19.9	9.111	1.822	23.9	8.434	1.687	27.9	7.842	1.568
16.0	9.870	1.974	20.0	9.092	1.818	24.0	8.418	1.684	28.0	7.828	1.566
16.1	9.849	1.970	20.1	9.074	1.815	24.1	8.403	1.681	28.1	7.814	1.563
16.2	9.829	1.966	20.2	9.056	1.811	24.2	8.387	1.677	28.2	7.800	1.560
16.3	9.808	1.962	20.3	9.039	1.808	24.3	8.371	1.674	28.3	7.786	1.557
16.4	9.787	1.957	20.4	9.021	1.804	24.4	8.356	1.671	28.4	7.773	1.555
16.5	9.767	1.953	20.5	9.003	1.801	24.5	8.340	1.668	28.5	7.759	1.552
16.6	9.746	1.949	20.6	8.985	1.797	24.6	8.325	1.665	28.6	7.745	1.549
16.7	9.726	1.945	20.7	8.968	1.794	24.7	8.309	1.662	28.7	7.732	1.546
16.8	9.705	1.941	20.8	8.950	1.790	24.8	8.294	1.659	28.8	7.718	1.544
16.9	9.685	1.937	20.9	8.932	1.786	24.9	8.279	1.656	28.9	7.705	1.541
17.0	9.665	1.933	21.0	8.915	1.783	25.0	8.263	1.653	29.0	7.691	1.538
17.1	9.645	1.929	21.1	8.898	1.780	25.1	8.248	1.650	29.1	7.678	1.536
17.2	9.625	1.925	21.2	8.880	1.776	25.2	8.233	1.647	29.2	7.664	1.533
17.3	9.605	1.921	21.3	8.863	1.773	25.3	8.218	1.644	29.3	7.651	1.530
17.4	9.585	1.917	21.4	8.846	1.769	25.4	8.203	1.641	29.4	7.638	1.528
17.5	9.565	1.913	21.5	8.829	1.766	25.5	8.188	1.638	29.5	7.625	1.525
17.6	9.545	1.909	21.6	8.812	1.762	25.6	8.173	1.635	29.6	7.611	1.522
17.7	9.526	1.905	21.7	8.794	1.759	25.7	8.158	1.632	29.7	7.598	1.520
17.8	9.506	1.901	21.8	8.777	1.755	25.8	8.143	1.629	29.8	7.585	1.517
17.9	9.486	1.897	21.9	8.761	1.752	25.9	8.128	1.626	29.9	7.572	1.514
18.0	9.467	1.893	22.0	8.744	1.749	26.0	8.114	1.623	30.0	7.559	1.512
18.1	9.448	1.890	22.1	8.727	1.745	26.1	8.099	1.620	30.1	7.546	1.509
18.2	9.428	1.886	22.2	8.710	1.742	26.2	8.084	1.617	30.2	7.533	1.507
18.3	9.409	1.882	22.3	8.693	1.739	26.3	8.070	1.614	30.3	7.520	1.504
18.4	9.390	1.878	22.4	8.677	1.735	26.4	8.055	1.611	30.4	7.507	1.501
18.5	9.371	1.874	22.5	8.660	1.732	26.5	8.040	1.608	30.5	7.494	1.499
18.6	9.352	1.870	22.6	8.644	1.729	26.6	8.026	1.605	30.6	7.481	1.496
18.7	9.333	1.867	22.7	8.627	1.725	26.7	8.012	1.602	30.7	7.468	1.494
18.8	9.314	1.863	22.8	8.611	1.722	26.8	7.997	1.599	30.8	7.456	1.491
18.9	9.295	1.859	22.9	8.595	1.719	26.9	7.983	1.597	30.9	7.443	1.489

Derived using the formula in Standard Methods for the Examination of Water and Wastewater, Page 4-101, 18th Edition, 1992

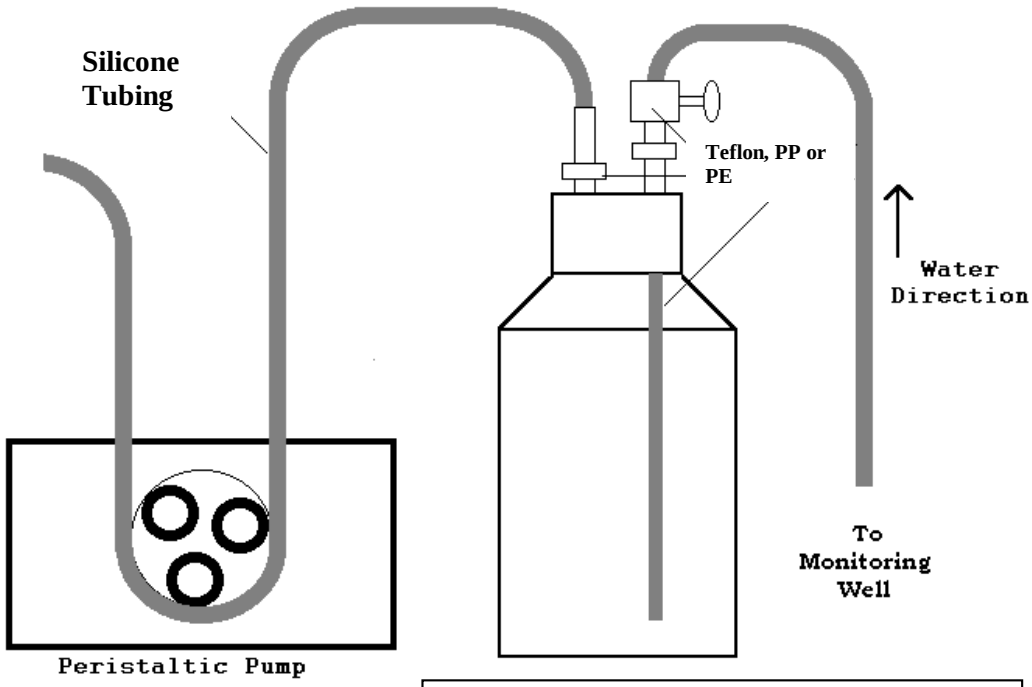
Table FS 2200-3
Allowable Uses for Bailers

• **ANALYTE GROUP(S)**	• **PURGING** (Not Recommended)	• **SAMPLING**	
	Use:	Use:	Not Recommended:
Volatile Organics Extractable Organics Radionuclides, including Radon Metals Volatile Sulfides	If allowed by permit, program, contract or order or If operated by a skilled individual with documented training in proper techniques. Field documentation must demonstrate that the procedure in FS 2213, section 4 was followed without deviation.	If concentrations exceed action levels, the purpose is to monitor effective treatment, and the DEP program allows the use of bailers; or If specified by DEP permit, program, contract or order. or If operated by a skilled individual with documented training in proper techniques and using appropriate equipment. Field documentation must demonstrate that the procedure in FS 2221, section 2 was followed without deviation.	If concentrations are near or below the stated action levels; or If a critical decision (e.g., clean closure) will be made based on the data; or If data are to demonstrate compliance with a permit or order.
Petroleum Hydrocarbons (TRPH) & Oil & Grease	If allowed by permit, program, contract or order or If operated by a skilled individual with documented training in proper techniques. Field documentation must demonstrate that the procedure in FS 2213, section 4 was followed without deviation.	Only if allowed by permit, program, contract or order as samples should be collected into the container without intermediate devices.	Unless allowed by permit, program, contract or order.

• **ANALYTE GROUP(S)**	• **PURGING** (Not Recommended)	• **SAMPLING**	
	Use:	Use:	Not Recommended:
Biologicals Inorganic Non-Metallics Aggregate Organics Microbiological Physical and Aggregate Properties	If allowed by permit, program, contract or order or If operated by a skilled individual with documented training in proper techniques. Field documentation must demonstrate that the procedure in FS 2213, section 4 was followed without deviation.	If all analytes collected from the well can be collected with a bailer; or If collected after collecting all analytes that require the use of a pump.	Before collecting any analytes that must be collected with a pump.
Ultra-Trace Metals	Never	Never	

Figure 2200-1

Pump and Trap for Extractable Organics



The glass sample bottle must be threaded to use a reusable sampling cap lined and installed with fittings made of Teflon, polypropylene or polyethylene, similar to the design shown.

Scenario 1: WELL SCREEN COMPLETELY SUBMERGED

Scenario 2: WELL SCREEN PARTIALLY SUBMERGED

Option 1a: Minimal Purge Volume: Pump or tubing is placed within the middle of the screen interval. The following conditions must be met to use this option:

1. The well screen interval is ≤ 10 feet.
2. Although drawdown may occur in the well when purging is initiated, the drawdown has to stabilize (Aquifer Recovery Rate = Purge Rate).
3. The samples will be obtained with the same equipment that was used to purge the well. Therefore, centrifugal pumps and bailers are not suitable for use in Option 1a.

If one or more of these conditions do not apply, use Option 1b.

Option 1b: Conventional Purge: Pump, tubing, or bailer [1] is placed above the screen at the top of the water column.

[1] DEP does not recommend the use of a bailer for purging; however, if a bailer is used it shall be lowered and raised at the rate of 2 cm/sec in the top of the water column.

Option 2a: A bailer [1] is placed at the top of the water column and is used to purge and sample the well.

Option 2b: Pump or tubing is placed within the middle of the saturated portion of the screen interval.

If the pump or tubing that was used for purging will not be used to obtain the sample, then position the pump or tubing at the top of the water column for purging.

Purging Procedure #1

1. After the drawdown in the well stabilizes, purge at least one equipment volume then collect the first set of stabilization parameters.
2. Thereafter, collect stabilization parameters ≥ 2 to 3 minutes apart.
3. Purge at least three equipment volumes before sampling.

Purging Procedure #2

1. Purge at least one well volume then collect first set of stabilization parameters.
2. Thereafter, collect stabilization parameters ≥ every 1/4 well volume.

Purging Procedure #3

1. Purge at least one well volume then collect first set of stabilization parameters.
2. Thereafter, collect stabilization parameters ≥ 2 to 3 minutes apart.

Purging Completion

If Dissolved Oxygen is ≤ 20% of saturation for the measured temperature and Turbidity is ≤ 20 NTUs, then purging is complete when three consecutive readings of the parameters listed below are within the following ranges:

Temperature ± 0.2 °C
pH ± 0.2 Standard Units
Specific Conductance ± 5.0% of reading

If Dissolved Oxygen (DO) is > 20% of saturation for the measured temperature and/or Turbidity is > 20 NTUs after every attempt has been made to reduce DO and/or turbidity, then purging is complete when three consecutive readings of the parameters listed below are within the following ranges:

Temperature ± 0.2 °C
pH ± 0.2 Standard Units
Specific Conductance ± 5.0% of reading
Dissolved Oxygen ± 0.2 mg/L or readings are within 10% (whichever is greater).
Turbidity ± 5 NTUs or readings are within 10% (whichever is greater).

If the well is expected to purge dry, position the pump or tubing within the screened interval and purge at ≤ 100 mL/minute until two equipment volumes are removed. Use the same pump for purging and sampling.

If the well purges dry at the lowest achievable flow rate (pumping at 100 mL/minute or less), then after a sufficient amount of water recharges in the well, collect the samples.

In either case listed above, before samples are collected, measure (once) pH, temperature, specific conductance, dissolved oxygen, and turbidity.

If one or more parameters do not stabilize after 5 volumes of the screened interval (purging procedure #1) or 5 well volumes (purging procedure #s 2 & 3) are removed, purging may be discontinued at the discretion of the sampling team leader.

FS 3000. SOIL

See also the following Standard Operating Procedures:

- FA 1000 Administrative Procedures
- FC 1000 Cleaning/Decontamination Procedures
- FD 1000 Documentation Procedures
- FM 1000 Field Planning and Mobilization
- FQ 1000 Field Quality Control Requirements
- FS 1000 General Sampling Procedures
- FT 1000 – FT 2000 Field Testing and Calibration

1. Introduction and Scope

1.1. Use these SOPs during field investigations to collect soil samples that are representative of current site conditions. It is very important to ensure that the collected samples are neither altered nor contaminated by sampling and handling techniques.

1.2. The following topics include: equipment choice, equipment construction materials, grab and areal or depth composite sampling techniques. Sample collection methods fall into three general depth classifications: surface, shallow subsurface, and deep subsurface. Once the samples are acquired, the handling procedures are very similar and are described below.

2. GENERAL

2.1. Select sampling equipment based on the type of sample to be collected and the analytes of interest. Choose soil sampling locations such that a representative portion of the soil is collected with minimal disturbance. Locations where natural vegetation is stressed or dead and/or areas that have surficial soil staining may be indicative of improper waste disposal practices.

2.2. If background and/or quality control sampling is warranted and feasible as determined in the site's work plan or by the project manager, select an up gradient, undisturbed location for obtaining the background and/or quality control samples. Be aware that differences in soil types may affect these background samples (e.g., sands vs. clays).

2.3. **Do not collect** samples for chemical analysis from auger flights or cuttings from hollow stem auger flights, except for waste characterization purposes for disposal.

2.4. Do not use samples that are collected for geological/lithological or vapor meter determinations for chemical analyses.

3. EQUIPMENT AND SUPPLIES

3.1. All equipment must be constructed of materials consistent with the analytes of interest. Refer to FS 1000, Tables FS 1000-1, FS 1000-2 and FS 1000-3 for selection of appropriate equipment and materials.

3.2. For information on sample container size and construction, see FS 1000, Table FS 1000-6.

3.3. For information on sampling equipment cleaning requirements, see FC 1000.

3.4. For information on preservation and holding time requirements, see FS 1000, Table FS 1000-6.

3.5. For information on documentation requirements, see FD 1000.

4. PROCEDURES FOR COMPOSITING

4.1. The following is not a complete discussion regarding all available sampling protocols nor the appropriateness or inappropriateness of compositing soil samples. The appropriateness of compositing soil samples will depend on the data quality objectives of the project. However, it is sometimes advantageous to composite soil samples to minimize the number of samples to be analyzed when sampling highly contaminated areas. Obtain permission from the DEP program.

4.1.1. Select sampling points from which to collect each aliquot.

4.1.2. Using the appropriate sampling technique, collect equal aliquots (same sample size) from each location and place in a properly cleaned container.

4.1.3. **Combine the aliquots of the sample directly in the sample container with no pre-mixing**.

4.1.4. Record the amount of each aliquot (volume or weight).

4.1.5. Label container, preserve on wet ice to 4°C and complete field notes.

4.1.6. Notify the laboratory that the sample is an unmixed composite sample, and request that the sample be thoroughly mixed before sample preparation or analysis.

5. SPECIFIC PROCEDURES FOR VOLATILE ORGANIC COMPOUNDS

Follow the procedures specified in EPA Method 5035 for sample collection and sample preparation. The protocols listed below **do not replace Method 5035** but clarify and/or modify certain method procedures. Therefore, it is essential that all organizations have a copy of Method 5035 as a reference document.

5.1. Container Preparation

5.1.1. All containers must be cleaned according to the FC 1000 sample container cleaning procedures for volatile organics.

5.1.2. Sample Vials: If sample vials are filled in the field, they must be provided with all reagents, stirring devices, label **and vial cap** to be used during sample analysis. These vials must be preweighed by the laboratory and records must be maintained so that there is an unambiguous link between the tare weight and the filled sample vial.

5.2. Collection Procedure

5.2.1. The sample vials (when used) will contain a premeasured amount of liquid. The laboratory must weigh the vials before sending into the field, and must weigh them again after receipt. Therefore:

- Do not lose any of the liquid either through evaporation or spillage
- Do not use a vial if some of the contents has spilled, or if it appears that some has leaked during transport
- Use the laboratory-supplied container label for identification information. **DO NOT apply any additional labels to the container**

- Do not interchange vial caps or septa

5.2.2. Minimize exposure to air by obtaining the sample directly from the sample source, using a coring device or a commercially designed sampling tool.

5.2.2.1. The sample collection device must be designed to fit tightly against the mouth of the vial or be small enough to be inserted into the vial. Use:

- EnCore or equivalent sampling devices or
- Disposable plastic syringes with the syringe end cut off prior to sampling (use **once** per sampling location).

5.2.2.2. Extrude the sample directly into the sample container.

5.2.3. Follow the method procedures for field transfer into the vial.

5.2.4. Procedures for determining the sample weight in the field are not required unless the project manager requires an accurate determination of the 5-gram sample size.

5.2.4.1. If the vials are returned to the laboratory for weighing, the sampler must be proficient in estimating the requisite 5-gram weight necessary for each sample.

5.2.4.2. If an accurate estimate of the 5-gram sample size is desired prior to starting sample collection activities, use a balance with a sensitivity of 0.1 gram. Check the balance calibration before each day's use with a set of weights that have been calibrated against NIST-traceable weights at least annually.

5.2.5. If the sampling device is transported to the laboratory with a sample, make sure the seals are intact, especially if collecting samples from sandy soils.

5.2.6. Collect at least two replicate samples from the same soil stratum and within close proximity to the original sample location.

5.2.7. Collect an additional aliquot of sample for screening and dry weight determinations.

5.3. Preservation (see FS 1000, Table FS 1000-7)

5.3.1. Low Level (≤ 200 µg/kg volatile organics)

5.3.1.1. Method 5035 discusses the use of sodium bisulfate, which is an acid. Since Florida soils contain significant amounts of calcium carbonate that reacts with acids, DEP does not recommend using this preservative.

5.3.1.2. Properly pack the samples (see FS 2004, section 5), and place all samples on wet ice.

5.3.1.3. Analyze unpreserved samples (no acid) within 48 hours.

5.3.1.4. Analyze acid-preserved samples within the specified 14-day holding time.

5.3.1.5. Analyze unpreserved samples that have been collected in a septum vial with premeasured analyte-free water within 48 hours.

5.3.1.6. If unpreserved samples collected in a septum vial with premeasured analyte-free water are frozen to -10°C at the laboratory within 48 hours of sample collection, analyze the samples within 14 days.

5.3.1.7. Analyze samples that have been collected with and transported in a sealed coring device within 48 hours.

5.3.1.8. If unpreserved samples collected in a sealed coring device are extruded from the corer into an appropriate liquid and frozen to -10°C at the laboratory within 48 hours of sample collection, analyze the samples within 14 days.

5.3.2. High Level (> 200 µg/kg volatile organics)

5.3.2.1. Properly pack the samples (see FS 2004, section 5), and place all samples on wet ice.

5.3.2.2. Analyze samples that have been collected with and transported in a sealed coring device within 48 hours.

5.3.2.3. If unpreserved samples collected in a sealed coring device are extruded from the corer into an appropriate liquid and stored at 4°C at the laboratory within 48 hours of sample collection, analyze the samples within 14 days.

5.3.2.4. Analyze samples that that have been preserved in methanol in the field within 14-days.

6. BULK SAMPLES: The collection of bulk samples will depend on the data quality objectives of the project.

6.1. Do not composite or mix VOC samples unless required by the DEP program or if mandated by a formal DEP document (permit, order or contract).

6.2. Select sampling points from which to collect each aliquot.

6.3. Using the appropriate sampling technique, collect equal aliquots (same sample size) from each location and place in a properly cleaned container.

6.3.1. **Combine the aliquots of the sample directly in the sample container with no pre-mixing**..

6.3.2. Pack soil tightly minimizing as much headspace as possible in the sample container.

6.3.3. Cap container tightly with Teflon side facing sample.

6.4. Record the amount of each aliquot (volume or weight) in the field notes.

6.5. Label container. Refer to FS 1000, Table FS 1000-7 for preservation and holding time requirements.

6.6. Notify the laboratory that the sample is an unmixed composite sample, and request that the sample be thoroughly mixed before sample preparation or analysis.

FS 3100. Surface Soil Sampling

Surface soil is generally classified as soil between the ground surface and 6-12 inches below ground surface.

1. Remove leaves, grass and surface debris from the area to be sampled.
2. Collect samples for volatile organic analyses as described in FS 3000, section 5.
3. Select an appropriate precleaned sampling device and collect the sample.
4. Transfer the sample to the appropriate sample container.
5. Clean the outside of the sample container to remove excess soil.

6. Label the sample container, place on wet ice to preserve to 4°C and complete the field notes.

FS 3200. Subsurface Soil Sampling

Interval begins at approximately 12 inches below ground surface.

FS 3210. SAMPLE COLLECTION PROCEDURE

Use the following after the desired depth has been reached by one of the methods outlined in FS 3220.

1. Collect samples for volatile organic analyses as described in FS 3000, section 5.

2. For other analyses, select an appropriate precleaned sampling device and collect the sample.

3. Transfer the sample to the appropriate sample container.

4. Clean the outside of the sample container to remove excess soil.

5. Label the sample container, place on wet ice to preserve to 4°C and complete the field notes.

FS 3220. REACHING THE APPROPRIATE DEPTH

1. SHOVELS AND DIGGERS: Used for soils from approximately 12 inches to a point when using the implement becomes impractical.

   1.1. Dig a hole or trench to the required depth.

   1.2. Follow the sample collection procedures outlined in FS 3210.

2. BACKHOE: Used for soils from approximately 12 inches to a point when using the implement becomes impractical.

   2.1. Dig a trench to the appropriate depth.

   2.2. Expose the sample, in the trench, by using a precleaned spoon, spatula or equivalent to clean away the soil that came in contact with the backhoe bucket.

   2.3. Use a **second** precleaned utensil to actually collect the sample from the trench.

   2.4. Follow the procedures outlined in FS 3210 to collect the sample.

3. BUCKET AUGERS AND HOLLOW CORERS: Suitable to reach soils from approximately 12 inches to a point when using the implement becomes impractical.

   3.1. Push and rotate the auger into the soil until the bucket is filled.

   3.2. Addition of a non-contaminating sleeve may allow an undisturbed soil sample to be obtained.

      3.2.1. The device consists of a standard auger head with a removable sleeve, which is inserted into the auger barrel. In this case it is the sleeve, which fills with soil.

      3.2.2. Remove the sleeve from the auger and cap.

   3.3. If the auger hole is prone to collapse due to low cohesion in some soils, DEP recommends inserting a temporary rigid PVC casing into the hole. The casing prevents hole collapse and minimizes cross-contamination between soil zones as the auger is advanced.

3.4. Remove the sample from the sampler by pushing or scraping the soil with an appropriate precleaned utensil into an appropriately precleaned tray or aluminum foil.

3.5. Remove any portion of the sample that has been disturbed and discard.

3.6. Follow the sample collection procedures outlined in FS 3210.

NOTE: If a confining layer has been breached during sampling, grout the hole to land surface with Type-1 Portland cement. This requirement may be different throughout Florida; contact the local Water Management District office for local requirements.

4. SPLIT SPOON SAMPLER: Suitable for reaching soils from approximately 12 inches to depths greater than 10 feet.

4.1. A split spoon sampler, useful for sampling unconsolidated soil, consists of two half cylinders (spoons) that fit together to form a tube approximately two feet in length and two inches in diameter.

4.1.1. The cylindrical arrangement is maintained by a retaining head and bit rings that screw on at each end of the split spoon.

4.1.2. The bit ring has beveled edges to facilitate sampling as the split spoon is forced into the ground.

4.1.3. Advance the sampler using the weight of the drilling stem and rods or a mechanical hammer.

4.1.4. Insert a catcher device in the head ring to prevent loss of unconsolidated sample during recovery.

4.2. After retrieving the split spoon sampler, expose the soil by unscrewing the bit and head rings and splitting the barrel.

4.3. If the recovery is enough to accommodate discarding a portion of the sample, discard the top and bottom two to three inches of the sample.

4.4. For volatile organic compounds collect the sample immediately from the **center portion of the split spoon** using the procedures described in FS 3000, section 5.

4.5. For other analyses, slice the sample from the center portion of the split spoon using a clean, decontaminated utensil.

4.6. Select an appropriate precleaned sampling device and collect the sample.

4.7. Transfer the sample to the appropriate sample container.

4.8. Clean the outside of the sample container to remove excess soil.

4.9. Label the sample container, place on wet ice to preserve to 4°C and complete the field notes.

5. DIRECT PUSH RIGS: May be used for depths greater than 10 feet below ground surface.

5.1. Liners: The clear liners are used with direct push rigs. This method is appropriate only for unconsolidated materials. The sampling depth that can be achieved varies depending on the rig and the lithologies that are encountered. Typically, the rig operator will:

- Place the liner inside the metal probe rod
- Select a point holder with an opening appropriate for the site lithology and screw it on the probe rod
- Advance the rod a full rod length
- Retrieve the rod
- Remove the point holder
- Remove the liner, and
- Slice the liner to expose the soil.

5.2. After the liner has been sliced, follow the procedures outlined in FS 3210, collecting volatile organic samples (if needed) immediately after the liner is sliced.

5.3. If samples for organic vapor analysis screening are required, collect them by slicing the sample(s) using a clean, decontaminated utensil and place them in 8-ounce (preferred) or 16-ounce jars, immediately cover the opening with aluminum foil and screw on the lid ring. If the contamination is derived from petroleum products, it is acceptable to use a clean gloved hand to transfer the sample(s) to the sample container(s).

5.4. For other analyses, slice the sample from the center portion of the split spoon using a clean, decontaminated utensil.

5.5. Select an appropriate precleaned sampling device and collect the sample.

5.6. Transfer the sample to the appropriate sample container.

5.7. Clean the outside of the sample container to remove excess soil.

5.8. Label the sample container, place on wet ice to preserve to 4°C and complete the field notes.

6. SHELBY TUBE SAMPLER

6.1. The Shelby tube sampler is used to sample unconsolidated soil and consists of a tube approximately 30 inches long and two inches (or larger) in diameter.

6.2. One end of the tube has edges beveled into a cutting edge. The other end can be mounted to an adapter, which allows attachment to the drilling rig assembly.

6.3. After drilling to the required depth with an auger or rotary drill bit, a soil sample is obtained through the auger or directly in the borehole.

6.4. Push the Shelby tube into the soil using the drilling rig's hydraulic ram or manually with a sledge hammer.

6.5. Remove the tube from the sampler head.

6.6. Extrude the sample from the Shelby tube.

6.7. Use a decontaminated utensil to remove any portion of the sample that has been disturbed.

6.8. Collect samples for volatile organics immediately from the center portion of the Shelby tube using the procedures described in FS 3000, section 5.

6.9. For other analyses, slice the sample from the center portion of the Shelby tube using a clean, decontaminated utensil.

6.10. Transfer the sample to the appropriate sample container.

6.11. Clean the outside of the sample container to remove excess soil.

6.12. Label the sample container, place on wet ice to preserve to 4°C and complete the field notes.

7. CORE BARREL

7.1. A standard core barrel is utilized when consolidated samples (such as limestone or dolomite) are to be sampled.

7.1.1. The core barrel is a cylinder approximately three feet long and two inches in diameter.

7.1.2. The barrel has a removable head ring with small embedded diamonds which allow the device to cut through rock or consolidated soil as the drilling rods are rotated.

7.2. Retrieve the sample core by unscrewing the head ring and sliding the sample into a precleaned container.

7.3. Use a decontaminated utensil to remove any portion of the sample that has been disturbed.

7.4. Remove the sample from the sampler (corer) with a precleaned tool.

7.5. Transfer the sample to the appropriate sample container.

7.6. Clean the outside of the sample container to remove excess soil.

7.7. Label the sample container, place on wet ice to preserve to 4°C and complete the field notes.

FT 1000. GENERAL FIELD TESTING AND MEASUREMENT

Use the following SOPs in conjunction with FT 1000:

- FD 1000 Documentation Procedures
- FM 1000 Field Planning and Mobilization
- FS 1000 General Sampling Procedures
- FT 1100 through FT 3000 Specific Field Testing Procedures

1. INTRODUCTION

1.1. Scope and Applicability: SOPs FT 1100 to FT 3000 outline procedures to conduct field testing measurements and observations. They include the parameters that are measured *in-situ* or in a field-collected sample. Additionally some samples with allowable extended holding times may be collected for laboratory measurement, as described in the specific FT-series SOPs. Included in SOPs FT 1100 to FT 3000 are:

- FT 1100 Field Measurement of Hydrogen Ion Activity (pH)
- FT 1200 Field Measurement of Specific Conductance (Conductivity)
- FT 1300 Field Measurement of Salinity
- FT 1400 Field Measurement of Temperature
- FT 1500 Field Measurement of Dissolved Oxygen (DO)
- FT 1600 Field Measurement of Turbidity
- FT 1700 Field Measurement of Light Penetration (Secchi Depth and Transparency)
- FT 1800 Field Measurement of Water Flow and Velocity
- FT 1900 Continuous Monitoring with Installed Meters
- FT 2000 Field Measurement of Residual Chlorine
- FT 3000 Aquatic Habitat Characterization

1.2. Exclusions: **If proposed for experimental purposes, field-screening procedures employing techniques not addressed in these SOPs** must be submitted to the DEP site or project manager. Such procedures must be addressed for each program or project dealing specifically with the planning and design of sampling events. Data quality objectives for quantitative assessment preclude the use of field-screening procedures for regulatory purposes.

1.3. Expectations and Requirements:

1.3.1. In some cases, specific instruments are identified in the SOP, with detailed instruction provided on their use. If you are using a different instrument from that identified in the SOP, follow the manufacturer's instructions for assembly, operation, and maintenance.

1.3.2. When required, the FT-series SOPs outline the instrument specifications. A field instrument must meet the stated requirements.

1.3.3. The FT-Series SOPs specify the calibration requirements for each method. Although instruments may vary in configuration or operation, the specified calibration requirements must be met.

1.3.3.1. Where applicable to the FT-series SOP, use the minimum number of calibration standards specified.

1.3.3.2. Do not establish the lower limit of the quantitative calibration bracket with "zero" solutions, quality control blanks or reagent dilution water.

1.3.4. Ensure that all equipment is in proper working condition, calibrated, and that batteries are properly charged before using the equipment for field testing measurements.

1.3.5. If reagents or standards are prepared from stock chemicals, they must be analytical reagent grade or better. Some procedures may specify a higher grade or assay of reagent or standard.

1.4. Recommendations for Use of Grab Samples or *in situ* Field Testing Measurements:

1.4.1. Use *in situ* readings where practical for field measurements in surface water and wastewater.

1.4.2. Use *in situ* readings or flow-through containers for field measurements for groundwater stabilization during purging and for other applications where groundwater monitoring measurements are required.

1.4.3. If grab samples are collected for measurement where allowed in the individual FT-series SOP, measure samples within fifteen (15) minutes of collection when immediate analysis is specified per Table FS 1000-4 and FS 1000-5. Otherwise, analyze grab samples within the applicable holding times specified in Table FS 1000-4 and FS 1000-5.

2. MINIMUM CALIBRATION REQUIREMENTS:

2.1. Calibration Definitions: This section outlines the essential calibration concepts that must be applied to each field test. Specific requirements for calibration are addressed in the individual SOPs.

2.1.1. Initial Calibration (IC): The instrument or meter electronics are adjusted (manually or automatically) to a theoretical value (e.g., dissolved oxygen saturation) or a known value of a calibration standard.

2.1.2. Initial Calibration Verification (ICV): The instrument or meter calibration is checked or verified directly following initial calibration by measuring a calibration standard of known value as if it were a sample and comparing the measured result to the calibration acceptance criteria listed in the SOP.

2.1.3. Continuing Calibration Verification (CCV): The instrument or meter calibration is checked or verified by measuring a calibration standard of known value as if it were a sample and comparing the measured result to the calibration acceptance criteria listed in the SOP.

2.1.4. Chronological Calibration Bracket: The interval of time between verifications within which environmental sample measurements must occur. The instrument or meter

is calibrated or verified before and verified after the time of environmental sample measurement(s).

2.1.5. Quantitative Calibration Bracket: The instrument or meter is calibrated or verified at two known values that encompass the range of observed environmental sample measurement(s).

2.1.6. Acceptance Criteria: The numerical limits within which calibration verifications are acceptable.

2.2. Calibration Activities: Specific calibration procedures are given in the individual SOPs.

2.2.1. Chronological Calibration Bracket:

2.2.1.1. Ensure that the field test result is preceded by an acceptable ICV or CCV and followed by an acceptable CCV.

2.2.1.2. Specific requirements for chronological bracketing are addressed in the individual FT-series SOPs.

2.2.2. Quantitative Calibration Bracket:

2.2.2.1. Choose two standards that bracket the range of sample measurements. These standards may be used for initial calibrations or for verifications.

2.2.2.2. Specific requirements for quantitative bracketing are addressed in the individual FT-series SOPs.

2.2.3. Initial Calibration: Calibrate if no initial calibration has been performed or if a calibration verification does not meet acceptance criteria. Do not reuse standards for initial calibrations.

Table FT 1000-1: Field Testing Acceptance Criteria

Parameter	Acceptance Criteria
pH (FT 1100)	**± 0.2 Standard pH Units of buffer or more stringent program criteria**
Specific Conductance (FT 1200)	**± 5% of standard value**
Temperature (FT 1400)	**± 0.2°C of NIST-traceable value (with correction factors) Verification over range of applicable values**
Dissolved Oxygen (FT 1500)	**± 0.3 mg/L of theoretical value (see Table FT 1500-1)**
Turbidity (FT 1600)	**0.1-10 NTU: ± 10% of standard value 11-40 NTU: ± 8% of standard value 41-100 NTU: ± 6.5% of standard value > 100 NTU: ± 5% of standard value**
Total Residual Chlorine (FT 2000)	**0.995 calibration curve correlation coefficient ± 10% of primary standard value ± 10% of secondary standard value Color comparator acceptance criterion: ± 10% of primary standard value**

2.2.4. Initial Calibration Verification:

2.2.4.1. Perform an ICV immediately after calibration. All ICVs must meet the calibration acceptance criteria specified in the applicable FT-series SOP. See Table FT 1000-1 for a list of acceptance criteria for the most common field testing procedures.

2.2.4.2. If an ICV fails to meet acceptance criteria, immediately recalibrate the instrument using the applicable initial calibration procedure or remove it from service.

2.2.5. Continuing Calibration Verification: Perform a CCV at no more than 24-hour intervals from previous verification, except where noted for individual FT-series SOPs.

2.2.5.1. If historically generated data demonstrate that a specific instrument remains stable for longer periods of time, the time interval between calibration verifications may be increased.

2.2.5.2. Base the selected time interval on the shortest interval that the instrument maintains stability. If CCVs consistently fail, shorten the time period between verifications or replace/repair the instrument.

2.2.5.3. All CCVs must meet the calibration acceptance criteria specified in the applicable FT-series SOP. See Table FT 1000-1 for a list of acceptance criteria for the most common field testing procedures.

2.2.5.4. If a CCV fails to meet acceptance criteria perform one or more of the following procedures as necessary:

- Reattempt the CCV again within the chronological bracket time interval without changing the instrument calibration. Do not perform maintenance, repair, or cleaning of the instrument or probe. Probes may be rinsed with analyte-free water or fresh verification standard. The CCV may be reattempted with a fresh aliquot of verification standard.
- Perform the initial calibration, perform an ICV, re-analyze the sample(s), and perform a CCV.
- Report all results between the last acceptable calibration verification and the failed calibration verification as estimated (report the value with a "J"). Include a narrative description of the problem in the field notes.

2.2.5.5. For installed instruments that are deployed for extended periods of time or used for continuous monitoring, see FT 1900.

2.2.5.6. Shorten the time period between verification checks or replace/repair the instrument.

2.2.6. Determining the Values of Secondary Standards: Use only those standards recommended by the manufacturer for a specific instrument. Only use secondary standards for continuing calibration verifications. See the individual FT-series SOPs for specific procedures for use of secondary standards. At documented intervals, determine or verify the values of secondary standards immediately after performing an initial calibration or after verifying the calibration with primary standards. Read each secondary standard as a sample. This result must be within the manufacturer's stated tolerance range and +/- 10% of the stated standard value. If the +/- 10% criterion is not

met, assign this reading as the value of the standard. If the reading is outside the manufacturer's stated tolerance range, discard the secondary standard.

2.2.7. **More frequent calibration verifications may be required for discharge permit compliance measurements or other regulatory requirements.**

3. PREVENTIVE MAINTENANCE: Record all maintenance and repair notes in the maintenance logbook for each meter (see FS 1007). If rental equipment is used, a log is not required. However, the origin (i.e., rental company), rental date, equipment type, model number, and identification number (if applicable) must be entered into the field notes or a rental equipment notebook.

4. DOCUMENTATION

4.1. Standard and Reagent Documentation: Document information about standards and reagents used for calibrations, verifications, and sample measurements.

4.1.1. Note the date of receipt, the expiration date and the date of first use for all standards and reagents.

4.1.1.1. Document acceptable verification of any standard used after its expiration date.

4.1.2. Record the concentration or other value for the standard in the appropriate measurement units.

4.1.2.1. Note vendor catalog number and description for pre-formulated solutions as well as for neat liquids and powdered standards.

4.1.2.2. Retain vendor assay specifications for standards as part of the calibration record.

4.1.3. Record the grade of standard or reagent used.

4.1.4. When formulated in-house, document all calculations used to formulate calibration standards.

4.1.4.1. Record the date of preparation for all in-house formulations.

4.1.5. Describe or cite the procedure(s) used to prepare any standards in-house (DEP SOP or internal SOP).

4.2. Field Instrument Calibration Documentation: Document acceptable calibration and calibration verification for each instrument unit and field test or analysis, linking this record with affected sample measurements.

4.2.1. Retain vendor certifications of all factory-calibrated instrumentation.

4.2.2. Designate the identity of specific instrumentation in the documentation with a unique description or code for each instrument unit used.

4.2.2.1. Record the manufacturer name, model number, and identifying number such as a serial number for each instrument unit.

4.2.3. Record the time and date of all initial calibrations and all calibration verifications.

4.2.4. Record the instrument reading (value in appropriate measurement units) of all calibration verifications.

4.2.5. Record the name of the analyst(s) performing the calibration.

4.2.6. Document the specific standards used to calibrate or verify the instrument or field test with the following information:

- Type of standard or standard name (e.g., pH buffer)
- Value of standard, including correct units (e.g., pH = 7.0 SU)
- Manufacturer's tolerance range for secondary standards
- Link to information recorded according to section 4.1 above

4.2.7. Retain manufacturers' instrument specifications.

4.2.8. Document whether successful initial calibration occurred.

4.2.9. Document whether each calibration verification passed or failed.

4.2.10. Document any corrective actions taken to correct instrument performance according to records requirements of FD 3000.

4.2.10.1. Document the date and time of any corrective actions.

4.2.10.2. Note any incidence of discontinuation of use of the instrument due to calibration failure.

4.2.11. Describe or cite the specific calibration or verification procedure performed (DEP SOP or internal SOP).

4.3. Record all field-testing measurement data, to include the following:

- Project name
- Date and time of measurement or test (including time zone, if applicable)
- Source and location of the measurement or test sample (e.g., monitoring well identification number, outfall number, station number or other description)
- Latitude and longitude of sampling source location (if required)
- Analyte or parameter measured
- Measurement or test sample value
- Reporting units
- Initials or name of analyst performing the measurement
- Unique identification of the specific instrument unit(s) used for the test(s)

Appendix FT 1000
Tables, Figures and Forms

Table FT 1000-1 Field Testing Acceptance Criteria

Table FT 1000-1: Field Testing Acceptance Criteria	
Parameter	**Acceptance Criteria**
pH (FT 1100)	**± 0.2 Standard pH Units of buffer or more stringent program criteria**
Specific Conductance (FT 1200)	**± 5% of standard value**
Temperature (FT 1400)	**± 0.2°C of NIST-traceable value (with correction factors) Verification over range of applicable values**
Dissolved Oxygen (FT 1500)	**± 0.3 mg/L of theoretical value (see Table FT 1500-1)**
Turbidity (FT 1600)	**0.1-10 NTU: ± 10% of standard value 11-40 NTU: ± 8% of standard value 41-100 NTU: ± 6.5% of standard value > 100 NTU: ± 5% of standard value**
Total Residual Chlorine (FT 2000)	**0.995 calibration curve correlation coefficient ± 10% of primary standard value ± 10% of secondary standard value Color comparator acceptance criterion: ± 10% of primary standard value**

FT 1100. Field Measurement of Hydrogen Ion Activity (pH)

Use in conjunction with:

- FT 1000 General Field Testing and Measurement
- FQ 1000 Field Quality Control Requirements
- FS 1000 General Sampling Procedures
- FD 1000 Documentation Procedures

1. Equipment and Supplies

1.1. Field Instrument: Use any pH meter consisting of a potentiometer, a glass electrode, a reference electrode, and a temperature-compensating device.

1.1.1. For routine fieldwork use a pH meter accurate and reproducible to at least 0.2-unit in the range of 0.0 to 14.0 units, and equipped with temperature-compensation adjustment. Record the pH value in pH units to one decimal place.

1.1.2. Advanced silicon chip pH sensors (with digital meters) may be used if demonstrated to yield equivalent performance to glass electrode sensors for the intended application.

1.2. Standards: Purchased or laboratory-prepared standard buffer solutions of pH values that bracket the expected sample pH range. Use buffers with nominal values of 4.0, 7.0 and 10.0 units for most situations. If the sample pH is outside the range of 4.0 to 10.0, then use two buffers that bracket the expected range with the pH 7 buffer being one of the two buffers. Alternatively, prepare appropriate standards per table I in method SM4500-H^+-B.

1.3. Recordkeeping and Documentation Supplies:

- Field notebook (w/ waterproof paper is recommended) or forms
- Indelible pens

2. Calibration and Use

2.1. General Concerns

2.1.1. The acceptance criterion for the initial calibration or the calibration verification is a reading of the standard within +/- 0.2-unit of the expected value.

2.1.2. On a weekly basis, check the calibration to ensure the % theoretical slope is greater than 90% (if applicable to your instrument type).

2.1.2.1. Note the % slope in the calibration records.

2.1.2.2. A % slope of less than 90% indicates a bad electrode that must be changed or repaired.

2.1.2.3. If % slope cannot be determined on your meter, or the manufacturer's optimum specifications are different, follow the manufacturer's recommendation for maintaining optimum meter performance.

2.2. Interferences

2.2.1. Sodium at pH $\geq$ 10.0 units can be reduced or eliminated by using a low sodium error electrode.

2.2.2. Coatings of oils, greases, and particles may impair the electrode's response. Pat the electrode bulb dry with lint-free paper or cloth and rinse with de-ionized water. For cleaning hard-to-remove films, use acetone very sparingly so that the electronic surface is not damaged.

2.2.3. Temperature effects on the electrometric measurement of pH are controlled by using instruments having temperature compensation or by calibrating the meter at the temperature of the samples.

2.2.4. Poorly buffered solutions with low specific conductance (< 200 μmhos/cm) may cause fluctuations in the pH readings. Equilibrate electrode by immersing in several aliquots of sample before taking pH.

2.2.5. Ensure stable sample and sensor temperature before calibrating or taking sample readings. Drifting sensor or sample temperature may produce erroneous sample measurements, calibrations, or verifications.

2.2.6. Thoroughly rinse the pH sensor with deionized water or fresh buffer standard when calibrating or verifying the calibration or when taking sample measurements. For in-situ measurements, ensure adequate flushing of the sensor with fresh sample water prior to taking measurements. Any residual standard, sample or deionized water remaining on the sensor may affect the measurement of the subsequent standard or sample. This is especially true when samples or standards of widely different pH value are successively measured.

2.2.7. Drifting readings or an inability to calibrate the sensor may also indicate a fouled electrode. Clean the electrode per the manufacturer's instructions or replace.

2.3. Calibration: Follow the manufacturer's calibration instructions specific to your meter. Most instruments allow for a two-point calibration and a few models can perform a three-point calibration. Use the appropriate number of standard buffer solutions for calibration. Do not reuse buffers for initial calibrations.

2.3.1. Rinse the probe with de-ionized water (DI) before and between each standard buffer solution.

2.3.2. Follow the calibration activities specified in FT 1000, section 2.2.

2.3.2.1. Perform an initial calibration using at least two buffers. Always use a pH 7 buffer first.

2.3.2.2. If the pH sample range is expected to be wider than the range established by a two-point calibration (e.g., some samples at pH 4 and others at pH 8), then add a third calibration point. If the instrument cannot be calibrated with three buffers, the third buffer may be used as the initial calibration verification to extend the range.

2.3.2.3. After initial calibration, immediately perform an initial calibration verification (ICV). Read a buffer as a sample. To be acceptable, a calibration verification must be within +/- 0.2 pH units of the stated buffer value. For example, if reading the pH 4.0 buffer, the result must be in the 3.8 to 4.2 range. Certain regulatory programs may have more stringent acceptance criteria.

2.3.2.4. After sample measurement(s), perform a continuing calibration verification (CCV). Read a buffer as a sample. To be acceptable, a

calibration verification must be within +/- 0.2 pH units of the stated buffer value. This CCV (if within acceptance criteria) can be used as the beginning of the chronological bracket. Certain regulatory programs may have more stringent acceptance criteria.

2.4. Measuring pH *in situ*: After calibrating the multi-probe sensors as outlined in 2.3 above, follow the meter's instructions to select the display for reading the pH of the sample. Immerse the probe at the desired depth in the water and wait for stabilization of the reading before recording the measurement.

2.5. Measuring pH in Flow-through Cells: When using a flow-through cell, the procedure described above in section 2.4 is applicable.

2.6. Measuring pH in Samples: After an acceptable initial calibration or calibration verification, follow these procedures to take a pH reading of a freshly collected sample (within 15 minutes of collection).

2.6.1. Pour enough of the fresh sample into a clean cup to take the reading.

2.6.2. Place the pH electrode in the sample (in the cup) and swirl the electrode.

2.6.3. Wait for stabilization, and read the pH value.

2.6.4. Turn the meter off after the last sample reading, rinse the electrode thoroughly with de-ionized water and replace the electrode's cap.

3. PREVENTIVE MAINTENANCE: Refer to FT 1000, section 3.

4. DOCUMENTATION

4.1. Standard and Reagent Documentation: Document information about standards and reagents used for calibrations, verifications, and sample measurements.

4.1.1. Note the date of receipt, the expiration date and the date of first use for all standards and reagents.

4.1.1.1. Document acceptable verification of any standard used after its expiration date.

4.1.2. Record the concentration or other value for the standard in the appropriate measurement units.

4.1.2.1. Note vendor catalog number and description for preformulated solutions as well as for neat liquids and powdered standards.

4.1.2.2. Retain vendor assay specifications for standards as part of the calibration record.

4.1.3. Record the grade of standard or reagent used.

4.1.4. When formulated in-house, document all calculations used to formulate calibration standards.

4.1.4.1. Record the date of preparation for all in-house formulations.

4.1.5. Describe or cite the procedure(s) used to prepare any standards in-house (DEP SOP or internal SOP).

4.2. Field Instrument Calibration Documentation: Document acceptable calibration and calibration verification for each instrument unit and field test or analysis, linking this record with affected sample measurements.

4.2.1. Retain vendor certifications of all factory-calibrated instrumentation.

4.2.2. Designate the identity of specific instrumentation in the documentation with a unique description or code for each instrument unit used.

4.2.2.1. Record manufacturer name, model number, and identifying number such as a serial number for each instrument unit.

4.2.3. Record the time and date of all initial calibrations and all calibration verifications.

4.2.4. Record the instrument reading (value in appropriate measurement units) of all calibration verifications.

4.2.5. Record the name of the analyst(s) performing the calibration.

4.2.6. Document the specific standards used to calibrate or verify the instrument or field test with the following information:

- Type of standard or standard name (e.g., pH buffer)
- Value of standard, including correct units (e.g., pH = 7.0 SU)
- Link to information recorded according to section 4.1 above

4.2.7. Retain manufacturers' instrument specifications.

4.2.8. Document whether successful initial calibration occurred.

4.2.9. Document whether each calibration verification passed or failed.

4.2.10. Document any corrective actions taken to correct instrument performance according to records requirements of FD 3000.

4.2.10.1. Document date and time of any corrective action.

4.2.10.2. Note any incidence of discontinuation of use of the instrument due to calibration failure.

4.2.11. Describe or cite the specific calibration or verification procedure performed (DEP SOP or internal SOP).

4.3. Record all field-testing measurement data, to include the following:

- Project name
- Date and time of measurement or test (including time zone, if applicable)
- Source and location of the measurement or test sample (e.g., monitoring well identification number, outfall number, station number or other description)
- Latitude and longitude of sampling source location (if required)
- Analyte or parameter measured
- Measurement or test sample value
- Reporting units
- Initials or name of analyst performing the measurement
- Unique identification of the specific instrument unit(s) used for the test(s)

FT 1200. Field Measurement of Specific Conductance (Conductivity)

Use in conjunction with:

- FT 1000 General Field Testing and Measurement
- FQ 1000 Field Quality Control Requirements
- FS 1000 General Sampling
- FD 1000 Documentation Procedures

1. INTRODUCTION: Specific conductance is a useful method to approximate the total amount of inorganic dissolved solids.

1.1. Conductivity varies with temperature. For example, the conductivity of salt water increases 3%/degree C at 0°C, and only 2%/degree C at 25°C.

1.2. Record the sample temperature or adjust the temperature of the samples prior to measuring specific conductance if the conductivity instrument does not employ automatic temperature compensation and correction of the instrument display value.

2. EQUIPMENT AND SUPPLIES

2.1. Field Instrument: Any self-contained conductivity instrument suitable for field work, accurate and reproducible to 5% or better over the operational range of the instrument, and preferably equipped with temperature-compensation adjustment. See references in FT 1210 below for additional information about instruments.

2.2. Standards: Purchased or laboratory-prepared standard potassium chloride (KCl) solutions with conductivity values that bracket the expected samples' range. In the laboratory, prepare standards of appropriate conductivities per SM2510 (Conductivity, in *Standard Methods for the Examination of Water and Wastewater, American Public Health Association*). Do not reuse standards for initial calibrations.

2.3. Recordkeeping and Documentation Supplies:

- Field notebook (w/ waterproof paper is recommended) or forms
- Indelible pens

3. CALIBRATION AND USE

3.1. General Concerns

3.1.1. Follow the instrument manufacturer's instructions for the details of operating the instrument.

3.1.2. For instruments without automatic temperature compensation, attempt to adjust the temperature of the samples to 25°C. If the temperature cannot be adjusted, measure the temperature with a calibrated device (see FT 1400), record the temperature, correct for temperature (per section 3.4 below) and report the results corrected to 25°C. See references in FT 1210 below for further information about temperature correction.

3.1.3. Ensure stable sample and sensor temperature before calibrating or taking sample readings. Drifting sensor or sample temperature may produce erroneous sample measurements, calibrations or verifications.

3.1.4. Thoroughly rinse the conductivity sensor with deionized water and fresh standard when calibrating or verifying the calibration or when taking sample measurements. For in-situ measurements, ensure adequate flushing of the sensor with fresh sample water prior to taking measurements. Any residual standard, sample or deionized water remaining on the sensor may affect the measurement of the subsequent standard or sample. This is especially true when samples or low-concentration standards are measured subsequent to measuring high-concentration standards.

3.1.5. Drifting readings or an inability to calibrate the sensor may also indicate a fouled electrode. Clean the electrodes per the manufacturer's instructions.

3.1.6. When successful calibration and verification cannot be achieved after ensuring that temperatures have stabilized and the sensor electrodes are clean and free of residual sample or standard from the previous measurement, suspect opened containers of standards, especially after repeated openings, when near the manufacturer's expiration date or when little standard volume remains in the container. Low-concentration conductivity standards are seldom stable for an extended period after opening.

3.2. Calibration and Calibration Verification:

3.2.1. Follow the calibration activities specified in FT 1000, section 2.2.

3.2.2. Initial Calibration: Calibrate the meter prior to use according to the following steps:

3.2.2.1. **Do not "zero" in the meter using analyte-free water or air.**

3.2.2.2. When the sample measurements are expected to be 100 μmhos/cm or greater, use two standard potassium chloride solutions that bracket the range of expected sample conductivities. A single standard at 100 μmhos/cm standard potassium chloride solution is acceptable for situations in which all sample measurements are expected to be less than 100 μmhos/cm.

3.2.2.3. Calibrate the instrument with one of the two standards to create an upper or lower boundary for the quantitative bracket.

3.2.2.4. Verify the calibration of the instrument with the second standard, quantitatively bracketing the range of expected sample values.

3.2.2.5. If the instrument can be calibrated with more than one standard, choose additional calibration standards within the range of expected sample values. The second standard in section 3.2.2.3 above may be used as an additional calibration standard.

3.2.2.6. Note: If all samples are expected to be less than 100 μmhos/cm, only one standard at 100 μmhos/cm standard potassium chloride solution is required.

3.2.3. Acceptability: Accept the calibration if the meter reads within +/- 5% of the value of any calibration standard used to verify the calibration. For example, the acceptance range for a 100 μmhos/cm standard is 95 to 105 μmhos/cm. If the meter does not read within +/- 5% of each calibration verification standard, determine the cause of the problem and correct before proceeding.

3.2.4. Temperature Correction: Most field instruments read conductivity directly. If the meter does not automatically correct values to 25°C, calculate correction factors using

the procedure in section 3.4 below. Record all readings and calculations in the calibration records.

3.2.5. Continuing Calibration Verification: Check the meter in read mode with at least one KCl standard with a specific conductance which quantitatively brackets the conductivity measured in environmental samples. The reading for the calibration verification must also be within +/- 5% of the standard value (see 3.2.3 above).

3.2.5.1. If new environmental samples are encountered outside the range of the initial calibration in 3.2.2 above, verify the instrument calibration with an additional standard that brackets the range of new sample values. If these calibration verifications fail, recalibrate the instrument as in 3.2.2.

3.2.5.2. **More frequent calibration verifications may be required for discharge permit compliance measurements or other regulatory requirements.**

3.3. Measuring Specific Conductance of Samples:

3.3.1. Follow manufacturer's instructions for sample measurement.

3.3.2. Immerse or place the conductivity probe or sensor in situ at a measuring location representative of the sampling source.

3.3.3. Allow the conductivity instrument to stabilize.

3.3.4. Measure the water temperature (if necessary for manual temperature compensation) and record the temperature. See FT 1400 for temperature measurement procedures.

3.3.5. If the meter is equipped with manual temperature compensation, adjust the conductivity meter to the water temperature per manufacturer's instructions.

3.3.6. If the conductivity meter has a set of positions that multiply the reading by powers of ten in order to measure the full range of potential conductivities, set this dial to the correct range in order to take a reading.

3.3.7. Record the sample conductivity measurement reading within 15 minutes of water sample collection.

3.3.8. Rinse off the probe with de-ionized water. Follow manufacturer's instructions for probe storage between use.

3.4 Calculations for Temperature Compensation

If the meter does not automatically correct for temperature (manual or automatic adjustment), or if a probe with a cell constant other than 1 is used, the following formula must be used to normalize the data to 25°C:

$$K = \frac{(K_m)\,(C)}{1 + 0.0191(T-25)}.$$

Where: K = conductivity in μmhos/cm at 25°C

K_m = measured conductivity in μmhos/cm at T degrees C

C = cell constant

T = measured temperature of the sample in degrees C

If the cell constant is 1, the formula for determining conductivity becomes:

$$K = \frac{(K_m)}{1 + 0.0191(T-25)}.$$

Refer to SM2510B, 20th edition, if other calculations (i.e., determining cell constant, etc.) are required. See FT 1210 below.

3.5 *In situ* Measurements at Depth or With Flow-through Cells: After calibrating the instrument as outlined in 3.2 above, follow the manufacturer's instructions to measure the conductivity of the sample.

3.5.1. For *in situ* measurements immerse the probe at the desired depth and wait for stabilization of the reading and record its value. Follow a similar procedure when using a flow-through cell.

3.5.1.1 Preferably measure groundwater sample conductivity *in situ* with a downhole probe or in a flow-through system.

4. PREVENTATIVE MAINTENANCE: Refer to FT 1000, section 3.

5. DOCUMENTATION

5.1. Standard and Reagent Documentation: Document information about standards and reagents used for calibrations, verifications and sample measurements.

5.1.1. Note the date of receipt, the expiration date and the date of first use for all standards and reagents.

5.1.1.1. Document acceptable verification of any standard used after its expiration date.

5.1.2. Record the concentration or other value for the standard in the appropriate measurement units.

5.1.2.1. Note vendor catalog number and description for preformulated solutions as well as for neat liquids and powdered standards.

5.1.2.2. Retain vendor assay specifications for standards as part of the calibration record.

5.1.3. Record the grade of standard or reagent used.

5.1.4. When formulated in-house, document all calculations used to formulate calibration standards.

5.1.4.1. Record the date of preparation for all in-house formulations.

5.1.5. Describe or cite the procedure(s) used to prepare any standards in-house (DEP SOP or internal SOP).

5.2. Field Instrument Calibration Documentation: Document acceptable calibration and calibration verification for each instrument unit and field test or analysis, linking this record with affected sample measurements.

5.2.1. Retain vendor certifications of all factory-calibrated instrumentation.

5.2.2. Designate the identity of specific instrumentation in the documentation with a unique description or code for each instrument unit used.

5.2.2.1. Record manufacturer name, model number, and identifying number such as a serial number for each instrument unit.

5.2.3. Record the time and date of all initial calibrations and all calibration verifications.

5.2.4. Record the instrument reading (value in appropriate measurement units) of all calibration verifications.

5.2.5. Record the name of the analyst(s) performing the calibration.

5.2.6. Document the specific standards used to calibrate or verify the instrument or field test with the following information:

- Type of standard or standard name (e.g., conductivity standard)
- Value of standard, including correct units (e.g., conductivity = 100 µmhos/cm)
- Link to information recorded according to section 5.1 above

5.2.7. Retain manufacturers' instrument specifications.

5.2.8. Document whether successful initial calibration occurred.

5.2.9. Document whether each calibration verification passed or failed.

5.2.10. Document any corrective actions taken to correct instrument performance according to records requirements of FD 3000.

5.2.10.1. Document date and time of any corrective action.

5.2.10.2. Note any incidence of discontinuation of use of the instrument due to calibration failure.

5.2.11. Describe or cite the specific calibration or verification procedure performed (DEP SOP or internal SOP).

5.3. Record all field-testing measurement data, to include the following:

- Project name
- Date and time of measurement or test (including time zone, if applicable)
- Source and location of the measurement or test sample (e.g., monitoring well identification number, outfall number, station number or other description)
- Latitude and longitude of sampling source location (if required)
- Analyte or parameter measured
- Measurement or test sample value
- Reporting units
- Initials or name of analyst performing the measurement
- Unique identification of the specific instrument unit(s) used for the test(s)

FT 1300. Field Measurement of Salinity

Use in conjunction with:

- FT 1000 General Field Testing and Measurement
- FQ 1000 Field Quality Control Requirements
- FS 1000 General Sampling Procedures
- FD 1000 Documentation Procedures

1. INTRODUCTION: Salinity is an important property of industrial and natural waters. This field parameter is also important for assessing the source or origin of effluents and of the mixing between fresh and marine waters in coastal regions, in both surface water and groundwater.

1.1. Salinity is a unit-less parameter since by definition it is the ratio of the mass of dissolved salts to the total mass of a given volume of water. Thus, salinity values are commonly expressed as "grams of salt/kilograms of water" or $^{o}/_{oo}$.

1.2. Salinity is determined by using indirect methods involving the measurement of a related physical property such as conductivity, density, sound speed, or refractive index. The commonly used procedures in the field are determination of conductivity or density of the sample.

1.3. The sample salinity is calculated from an empirical relationship between salinity and the physical property as determined from a standard solution. Refer to the referenced method SM2520 for further discussions on these topics.

1.4. Because of its high sensitivity and easy of measurement, the conductivity method is most often used to determine the salinity. (Note – using a hydrometer to measure the density or the specific gravity to obtain an approximate salinity value is not recommended for reporting purposes.)

2. EQUIPMENT AND SUPPLIES

2.1. Field Instrument: Depending on the chosen method, use:

2.1.1. Any self-contained conductivity instrument with a platinum or graphite electrode type cell, and a temperature sensor. Some conductivity instruments have meter scales pre-calibrated for salinity and are sometimes referred to as Salinometers. For routine fieldwork use a conductivity meter accurate and reproducible to at least 5% or 1 μmho/cm (whichever is greater), and equipped with temperature-compensation adjustment; or

2.1.2. A precision "vibrating flow densimeter" (see Millero & Poisson, 1981) and a field thermometer.

2.2. Standards:

2.2.1. Purchased or laboratory-prepared Standard Seawater and/or potassium chloride (KCl) standards of appropriate equivalent salinities.

2.2.1.1. In the laboratory, prepare the Standard Seawater per recipe in method SM2520 and SM8010 (Table III), and standard KCl solutions per recipe in method SM2510 (American Public Health Association, American Water Works Association, Water Pollution Control Federation, *Standard Methods for the Examination of Water and Wastewater)*.

2.2.2. De-ionized water for calibration of the densimeter (if used).

2.3. Recordkeeping and Documentation Supplies:

- Field logbook (w/ waterproof paper is recommended) or field forms
- Indelible pens

3. CALIBRATION AND USE

3.1. Conductivity Method

3.1.1. Calibration: - Calibrate the instrument per manufacturer's instructions using one calibration standard, either standard seawater or a KCl solution, as applicable. The acceptance criterion for initial calibration or a calibration verification is that the instrument reading is within +/- 5% of the standard value. For example, when calibrating with standard seawater, S = 35, the meter must read in the 34 to 36 range in order to be acceptable.

3.1.1.1. Use standard seawater (S = 35) when measuring salinity in the open ocean or estuaries with a predominance of seawater.

3.1.1.2. KCl may be used in estuarine waters with low salinity (S = 0 – 40).

3.1.1.3. If verifying or calibrating with a "zero" standard, do not use analyte-free water or air check (dry electrode) as the blank.

3.1.1.4. If the meter does not provide a direct reading of salinity, use the equation found in SM2520B to convert the readings to salinity.

3.1.1.5. Follow the calibration activities in FT 1000, section 2.2.

3.1.1.6. Do not reuse standards for initial calibrations.

3.1.2. Field Use: - Rinse the probe with DI water after calibration and before each sample measurements. Follow the manufacturer's instructions for temperature compensation, if needed. Report salinities with only one decimal figure.

3.1.3. General Concerns for Conductivity Method

3.1.3.1. Ensure stable sample and sensor temperature before calibrating or taking sample readings. Drifting sensor or sample temperature may produce erroneous sample measurements, calibrations, or verifications.

3.1.3.2. Thoroughly rinse the conductivity (salinity) sensor with deionized water and fresh standard when calibrating or verifying the calibration or when taking sample measurements. For in-situ measurements, ensure adequate flushing of the sensor with fresh sample water prior to taking measurements. Any residual standard, sample, or deionized water remaining on the sensor may affect the measurement of the subsequent standard or sample. This is especially true when samples or low-concentration standards are measured subsequent to measuring high-concentration standards.

3.1.3.3. Drifting readings or an inability to calibrate the sensor may also indicate a fouled electrode. Clean the electrodes per the manufacturer's instructions.

3.1.3.4. When successful calibration and verification cannot be achieved after ensuring that temperatures have stabilized and the sensor electrodes are clean and free of residual sample or standard from the previous measurement, suspect opened containers of standards, especially after repeated openings, when near the

manufacturer's expiration date or when little standard volume remains in the container. Low-concentration conductivity standards are seldom stable for an extended period after opening.

3.2. Density Method

The vibrating flow densimeter is an instrument that allows for precise and rapid measurements of the density of a liquid, such as water. The principle of operation is the effect of the density of the sample on the frequency of a vibrating tube encased in a constant-temperature jacket. The measurement is made by passing the water (sample) through the vibrating tube and reading the period of vibration that is electronically sensed and displayed by the densimeter. The sample density (D) is proportional to the square of the period of vibration (T):

$$D = a + bT^2$$

Where a and b are terms determined by calibration, b being determined by calibration of the densimeter with Standard Seawater. The difference between the density of the sample (D) and that of pure water (D_0) is given by:

$$D - D_0 = b\,(T^2 - T_0^2)$$

Where T and T_0 are, respectively, the periods of the sample and that of pure (de-ionized) water. Using this second equation, you only have to deal with the term b for calibration purposes. Hence, the system can be calibrated with two liquids: pure water and Standard Seawater. Follow the manufacturer's instruction for calibration of the densimeter.

The salinity of the sample is determined by the one-atmosphere international equation of state for seawater. This equation relates the difference ($D - D_0$) to the practical salinity as a function of the temperature of the sample (which is also measured by the densimeter or the field thermometer). For further details on this calculation read the referenced method SM2520C.

4. PREVENTIVE MAINTENANCE: Refer to FT 1000, section 3.

5. DOCUMENTATION

5.1. Standard and Reagent Documentation: Document information about standards and reagents used for calibrations, verifications, and sample measurements.

5.1.1. Note the date of receipt, the expiration date and the date of first use for all standards and reagents.

5.1.1.1. Document acceptable verification of any standard used after its expiration date.

5.1.2. Record the concentration or other value for the standard in the appropriate measurement units.

5.1.2.1. Note vendor catalog number and description for preformulated solutions as well as for neat liquids and powdered standards.

5.1.2.2. Retain vendor assay specifications for standards as part of the calibration record.

5.1.3. Record the grade of standard or reagent used.

5.1.4. When formulated in-house, document all calculations used to formulate calibration standards.

5.1.4.1. Record the date of preparation for all in-house formulations.

5.1.5. Describe or cite the procedure(s) used to prepare any standards in-house (DEP SOP or internal SOP).

5.2. Field Instrument Calibration Documentation: Document acceptable calibration and calibration verification for each instrument unit and field test or analysis, linking this record with affected sample measurements.

5.2.1. Retain vendor certifications of all factory-calibrated instrumentation.

5.2.2. Designate the identity of specific instrumentation in the documentation with a unique description or code for each instrument unit used.

5.2.2.1. Record manufacturer name, model number, and identifying number such as a serial number for each instrument unit.

5.2.3. Record the time and date of all initial calibrations and all calibration verifications.

5.2.4. Record the instrument reading (value in appropriate measurement units) of all calibration verifications.

5.2.5. Record the name of the analyst(s) performing the calibration.

5.2.6. Document the specific standards used to calibrate or verify the instrument or field test with the following information:

- Type of standard or standard name (e.g., salinity standard)
- Value of standard, including correct units (e.g., salinity = 20 $^{o}/_{oo}$)
- Link to information recorded according to section 5.1 above

5.2.7. Retain manufacturers' instrument specifications.

5.2.8. Document whether successful initial calibration occurred.

5.2.9. Document whether each calibration verification passed or failed.

5.2.10. Document any corrective actions taken to correct instrument performance according to records requirements of FD 3000.

5.2.10.1. Document date and time of any corrective action.

5.2.10.2. Note any incidence of discontinuation of use of the instrument due to calibration failure.

5.2.11. Describe or cite the specific calibration or verification procedure performed (DEP SOP or internal SOP).

5.3. Record all field-testing measurement data, to include the following:

- Project name
- Date and time of measurement or test (including time zone, if applicable)
- Source and location of the measurement or test sample (e.g., monitoring well identification number, outfall number, station number or other description)
- Latitude and longitude of sampling source location (if required)

- Analyte or parameter measured
- Measurement or test sample value
- Reporting units
- Initials or name of analyst performing the measurement
- Unique identification of the specific instrument unit(s) used for the test(s)

FT 1400. Field Measurement of Temperature

The use of this SOP is not required when using field temperature measurement devices to monitor groundwater stabilization during the purging of groundwater monitoring wells. Field temperature measurement devices used for temperature compensation (correction) for other measurements such as dissolved oxygen, specific conductance or pH are also exempted from the requirements of this SOP. FT 1400 must be used for all other field temperature measurements required by DEP.

Use this SOP in conjunction with the following DEP SOPs:

- FT 1000 General Field Testing and Measurement
- FQ 1000 Field Quality Control Requirements
- FS 1000 General Sampling Procedures
- FD 1000 Documentation Procedures

1. EQUIPMENT AND SUPPLIES

1.1. Field Instruments: Use any of the following instrument types for performing field measurements:

- Digital thermistor (thermocouple type) and meter typical of field instruments
- Glass bulb, mercury-filled thermometer (not recommended for field ruggedness)
- Glass bulb, alcohol-filled thermometer with protective case
- Bi-metal strip/dial-type thermometer
- Advanced silicon chip temperature sensor and digital meter

1.1.1. Field instruments must be capable of measuring temperature in 0.1^{o}C increments.

1.2. Standard Thermometer: NIST-traceable Celsius certified thermometer with scale marks for every 0.1^{o}C increment, a range of 0^{o}C to 100^{o}C (or a range bracketing expected sample temperatures) and correction chart supplied with certification. The standard thermometer must have a valid certification for the period of measurement.

1.3. Recordkeeping and Documentation Supplies:

- Field notebook or forms \
- Indelible pens

2. CALIBRATION AND USE

2.1. General Concerns

2.1.1. Select a temperature measuring device meeting the requirements of section 1.1 above.

2.1.2. Dial-type and thermocouple-type devices with meters are preferred over the glass thermometers for fieldwork because of their durability and ease of reading.

2.1.2.1. Transport glass thermometers in protective cases.

2.1.2.2. Inspect glass thermometers for liquid separation. Do not use a thermometer if the liquid has separated.

2.1.2.3. Most instruments with digital display will provide more decimal figures than are significant. Record the temperature reading with only one rounded decimal figure (e.g., 25.9 instead of 25.86^{o}C).

2.2. Calibration

2.2.1. Follow the calibration activities specified in FT 1000, section 2.2.

2.2.2. Verify all thermistor (meter) devices and field thermometers against the NIST-traceable standard thermometer at several temperatures in the expected sample measurement range, using any correction factor indicated by the certificate supplied with the NIST-traceable thermometer.

2.2.2.1. See the US Geological Survey, National Field Manual for the Collection of Water-Quality Data, Book 9, Chapter A6, Field Measurements, Section 6.1, Temperature, Techniques of Water-Resources Investigations, 4/98 for additional guidance about making temperature comparisons with the standard thermometer.

2.2.2.2. Make note of the calibration in the calibration records. See section 4 below.

2.2.2.3. The field measurement device may be used with a linear correction factor provided that the observed temperature difference with the standard thermometer is documented at incremental temperatures over the range of expected sample temperatures.

2.2.2.4. Use the resulting correction factor when making temperature measurements of samples with the field measurement device.

2.2.2.5. Prominently display the correction factor on the field measurement device, with the date last verified. A calibration correction curve or plot may also be used.

2.2.2.6 To be acceptable, a calibration verification must be within +/- 0.5^{o}C of the corrected reading of the NIST-traceable thermometer.

2.2.2.7 Properly dispose of glass-bulb thermometers that do not meet the above calibration acceptance criteria.

2.2.3. Continuing Calibration Verifications:

2.2.3.1. Determine the maximum time between continuing calibration verifications for the specific field temperature measurement device based on instrument stability.

2.2.3.2. Verify the field measurement device against the standard NIST-traceable thermometer as in section 2.2.2 above.

2.2.4. Refer to additional calibration requirements in FT 1000, section 2.2.

2.2.5. **More frequent calibration verifications may be required for discharge permit compliance measurements or other regulatory requirements.**

2.3. Measuring Sample Temperature

2.3.1. Insert or place the thermometer or sensor *in situ* at a measuring location representative of the sampling source.

2.3.2. Allow the thermometer or temperature sensor to equilibrate to ambient *in situ* temperature.

2.3.2.1. Groundwater samples must be measured *in situ* with a downhole probe or in a flow-through container. Do not measure bailed or pumped samples in an intermediate container containing static sample.

2.3.3. Record the temperature to the nearest 0.1°C after the reading stabilizes and remains constant.

3. PREVENTIVE MAINTENANCE: Refer to FT 1000, section 3.

4. DOCUMENTATION

4.1. Standards Documentation: Document information about the NIST-traceable standard thermometer in the calibration record, including:

- Unique identification for the thermometer
- Vendor certificate of calibration, including any correction factor
- Vendor's expiration date for the certificate of calibration

4.2. Field Instrument Calibration Documentation: Document acceptable calibration and calibration verification for each instrument unit and field test or analysis, linking this record with affected sample measurements.

4.2.1. Retain vendor certifications of all factory-calibrated instrumentation.

4.2.2. Designate the identity of specific instrumentation in the documentation with a unique description or code for each instrument unit used.

4.2.2.1. Record manufacturer name, model number, and identifying number such as a serial number for each instrument unit.

4.2.3. Record the time and date of all initial calibrations and all calibration verifications.

4.2.4. Record the instrument reading (value in appropriate measurement units) of all calibration verifications.

4.2.5. Record the name of the analyst(s) performing the calibration.

4.2.6. Document the following information about initial calibration and calibration verifications and link to information recorded according to section 4.1 above:

- Details of the method used to compare the field measurement device to the NIST-traceable standard thermometer.
- Results of each calibration verification, including the expected reading (per the NIST-traceable standard thermometer)
- The actual reading of the field measurement device, using any established correction factors and correct units.

4.2.7. Retain manufacturers' instrument specifications.

4.2.8. Document whether successful initial calibration occurred.

4.2.9. Document whether each calibration verification passed or failed.

4.2.10. Document any corrective actions taken to correct instrument performance (such as a new correction factor) according to records requirements of FD 3000.

4.2.10.1. Document date and time of any corrective action.

4.2.10.2. Note any incidence of discontinuation of use of the instrument due to calibration failure.

4.2.11. Describe or cite the specific calibration or verification procedure performed (DEP SOP or internal SOP).

4.3. Record all field-testing measurement data, to include the following:

- Project name
- Date and time of measurement or test (including time zone, if applicable)
- Source and location of the measurement or test sample (e.g., monitoring well identification number, outfall number, station number or other description)
- Latitude and longitude of sampling source location (if required)
- Analyte or parameter measured
- Measurement or test sample value
- Reporting units
- Initials or name of analyst performing the measurement
- Unique identification of the specific instrument unit(s) used for the test(s)

FT 1500. Field Measurement of Dissolved Oxygen (DO)

Use in conjunction with:

- FT 1000 General Field Testing and Measurement
- FS 1000 General Sampling Procedures
- FD 1000 Documentation Procedures

1. EQUIPMENT AND SUPPLIES

1.1. Field Instruments

1.1.1. Membrane-type polarographic or galvanic electrode DO sensor with dedicated meter or configured with multi-parameter sonde

1.1.2. Luminescence-based DO sensor with dedicated meter or configured with multi-parameter sonde (see American Society for Testing and Materials, *Standard Test Methods for Dissolved Oxygen in Water*, Test Method C-Luminescence-based Sensor, D 888-05).

1.1.3. Select instrument assemblies that provide minimum precision of +/- 0.2 mg DO/L and a minimum accuracy of +/- 0.2 mg DO/L.

1.1.4. Compensate for temperature dependence of DO measurements by using instruments employing automatic temperature compensation or by manually correcting measurements in accordance with SM 4500-O G (see *Standard Methods for the Examination of Water and Wastewater*, American Public Health Association, American Water Works Association, Water Pollution Control Federation).

1.1.4.1. Calibrate on-board temperature sensors as described in FT 1400.

1.2. Standards

1.2.1. NIST-traceable Celsius thermometer with a scale marked for every 0.1^{o}C and a range of 0 to 100^{o}C.

1.2.2. Access to an organization with capability to perform the Winkler titration procedure is recommended but not mandatory.

1.2.3. A “zero-DO standard”, prepared on-site with an aliquot of the sample water, is optional. Prepare by adding excess sodium sulfite and a trace of cobalt chloride to bring the DO to zero.

1.3. Recordkeeping and Documentation Supplies:

- Field notebook (w/ waterproof paper is recommended) or forms
- Indelible pens

2. CALIBRATION AND USE: the electrode method is predominantly used *in-situ* for dissolved oxygen determinations.

2.1. General Concerns

2.1.1. Turbulence is necessary to keep a constant flow of water across the membrane-sample interface. Make sure the appropriate mechanism is working before using the probe.

2.1.2. Follow instrument manufacturer's instructions for probe storage. For example, store the probe with a cover that creates a saturated atmosphere. A cap, with a wet sponge in it, will suffice for single-parameter probes. If the sensor is in a multi-probe device, keep the protective cap chamber moist during storage.

2.1.3. Before mobilizing, check to make sure there are no bubbles beneath the probe membrane, or any wrinkles or tears in the probe membrane. If so, replace the membrane and KCL solution. Check the leads, contacts, etc. for corrosion and/or shorts if meter pointer remains off-scale, does not calibrate, or drifts.

2.1.4. Dissolved inorganic salts interfere with the performance of DO probes. For example, DO readings in salt water are affected by the salinity and must be corrected. The DO meter may adjust automatically based on readings taken from the specific conductivity/salinity probe. If corrections are not automatic the appropriate calculations must be used to correct for salinity. If automatic adjustments are used the specific conductivity/salinity probe calibration must be verified or calibrated in accordance with FT1200.

2.1.5. Reactive gases, which pass through the membrane, may interfere. For example, chlorine will depolarize the cathode and cause a high probe output. Long-term exposures to chlorine will coat the anode with the chloride of the anode metal and eventually desensitize the probe. Sulfide (from H_2S) will undergo oxidation if high enough potential (voltage) is applied, creating current flow, yielding faulty readings. If such interferences are suspected, change the membrane electrode more frequently and calibrate at more frequent intervals.

2.1.6. Ensure that the temperature of the sensor and sample are stable. Unstable temperatures will produce erroneous calibrations, verifications or sample measurements.

2.1.7. Erroneous calibrations or verifications may result if the saturated air chamber is not vented to atmospheric pressure, properly humidified and protected from temperature fluctuations produced by common field conditions such as evaporation or fluctuation in sunlight intensity.

2.2. Follow the quality control requirements for calibration (see activities in FT 1000, section 2.2).

2.3. Initial Calibration and Initial Calibration Verification

2.3.1. Air Calibration and Initial Calibration Verification (ICV): Calibrate the meter at 100% saturation. Before use, verify the meter calibration in water-saturated air to make sure it is properly calibrated and operating correctly. Make a similar verification at the end of the day or sampling event. Follow the manufacturer's instructions for your specific instrument.

2.3.1.1. Allow an appropriate warm up period before initial field calibration.

2.3.1.2. Wet the inside of the calibration chamber with water, pour out the excess water (leave a few drops), wipe any droplets off the membrane/sensor and insert the sensor into the chamber (this ensures 100% humidity).

2.3.1.3. Allow adequate time for the DO sensor and the air inside the calibration chamber to equilibrate.

2.3.1.4. Once the probe/calibration chamber is stable at ambient temperature, check the air temperature and determine, from the DO versus temperature table, what the DO saturation value should be at the observed temperature (see Table FT

1500-1, below). A stable and accurate temperature is required for a valid calibration. The acceptance criterion for DO calibration verification is +/- 0.3 mg DO/L at the observed temperature of the verification.

2.4. Continuous Calibration Verification

2.4.1. Air-Calibration Verification: DO sensor or instrument is calibrated against air that is saturated with water at a known temperature and ambient atmospheric pressure. Use Table FT 1500-1 below to verify calibration at specified temperature.

2.4.1.1. Wet the inside of the calibration chamber with water, pour out the excess water (leave a few drops) and insert the sensor into the chamber (this ensures 100-percent humidity)

2.4.1.2. Allow adequate time for the DO sensor and the air inside the calibration chamber to equilibrate.

2.4.1.3. Measure the temperature in the calibration chamber and observe the readings until the instrument stabilizes.

2.4.1.4. Use the oxygen solubility Table FT 1500-1 below to determine the DO saturation at a measured temperature and atmospheric pressure. Calculate values to the nearest tenth degree by interpolation or use an expanded version of this table found in FS 2200, which provides saturation data in 0.1 ^{o}C increments for a selected temperature range (see Table FS 2200-2).

2.4.1.5. Compare DO meter reading with value obtained from Table FT 1500-1 below to verify continuous calibration.

2.5. Additional Verifications: The following methods may be used as additional checks to verify calibration. These additional checks may be required as part of a specific permit.

2.5.1. Winkler method: This check is useful to assess the condition of the DO sensor (i.e., its degradation with time/use) and that the instrument can still maintain a valid calibration (see SM 4500-O C).

2.5.1.1. **Perform the Winkler method when required by permit or other regulation at the required calendar frequency.**

2.5.1.2. For an accuracy calibration verification using the Winkler method, follow SM 4500-O C.

2.5.1.3. Fill a clean bucket with uncontaminated or de-ionized water and place the probe into the bucket (with stirrer or equivalent mechanism turned off). Fill at least two biological oxygen demand (BOD) bottles without entraining atmospheric oxygen into the bottles. Carefully submerge the bottom of the bottle (one at a time) into the water and allow the water to fill the bottle. Place the bottle on the bottom of the bucket and carefully place stopper into it without adding atmospheric oxygen. Retrieve the bottles and determine their DO by the Winkler method (see SM4500-O-C for more details). Turn the stirrer or equivalent mechanism on and read the DO of the water in the bucket.

2.5.1.4. Adjust the DO meter according to manufacturer's instructions. Be sure to adjust the meter to the temperature of water in the bucket, and then calibrate the DO meter to read the average DO concentration of the two samples determined by the Winkler test.

2.5.2. Zero-DO Verification: The air calibration and the interfering effects of the sample can be further checked in the field by means of a "zero-DO standard"(SM 4500-O G).

2.5.2.1. Prepare this standard on-site with an aliquot of the sample by adding excess sodium sulfite and a trace of cobalt chloride to bring the DO to zero. Prepare this zero-DO standard in a beaker or a large-mouth sample container of appropriate size to insert the DO probe.

2.5.2.2. After adding the chemicals, gently swirl the water and let it sit for about 30 seconds before inserting the probe.

2.5.2.3. Read the DO of the sample. If the reading is outside the acceptance interval, the instrument must be recalibrated and/or zero-adjusted if the meter allows for this adjustment.

2.5.3. Air-Saturated Water: The DO sensor or instrument system is calibrated against water that is saturated with oxygen at a known temperature and ambient atmospheric pressure.

2.5.3.1. The temperature and conductivity of water used for calibration should be about the same as the temperature and conductivity of the water to be measured.

2.5.3.2. Place DO sensor and calibration water in a large beaker or open-mouth container.

2.5.3.3. Aerate the water for an adequate amount of time.

2.5.3.4. Determine if the water is 100 percent saturated with oxygen, and take a temperature reading. Temperature must be calibrated or verified for accuracy before DO calibration verification.

2.5.3.5. Use Table FT 1500-1 above to determine the DO saturation value at the measured water temperature. Compare DO meter reading with value obtained from Table FT 1500-1 to ensure continuous calibration.

2.6. Measuring DO in Samples:

2.6.1. Insert or place the DO probe *in situ* at a measuring location representative of the sampling source:

2.6.1.1. Take the DO of an effluent just before it enters the receiving water. If the effluent aerated prior to entering the surface water, take the DO reading in the receiving water right where it enters.

2.6.1.2. For well mixed surface waters, e.g., fast flowing streams, take the DO reading at approximately 1-2 feet below the surface or at mid-depth.

2.6.1.3. For still or sluggish surface waters, take a reading at one foot below the surface, one foot above the bottom, and at mid-depth.

2.6.1.4. If it is shallow surface waters, (less than two feet) take the reading at mid-depth.

2.6.1.5. Do not take a reading in frothy or aerated water unless required by the sampling plan.

2.6.1.6. Groundwater samples must be measured *in situ* with a downhole probe or in a flow-through container. Do not measure bailed or pumped samples in an intermediate container containing static sample.

2.6.2. Rinse probe with de-ionized water and keep the probe in the saturated atmosphere (see 2.1.2 above) between sites and events.

2.6.3. If the readings show distinct, unexplainable changes in DO levels, or when the probe has been in waters with high sulfides, recalibrate or perform maintenance per manufacturer's instructions. While taking a reading, if it is very low (e.g., below 1.0 mg/L), allow the meter to stabilize, record it and then, remove and rinse the probe, as the environment is very likely anoxic and may contain hydrogen sulfide, which can damage the probe.

2.6.4. Salinity and Temperature corrections may be necessary. Follow manufacturer instructions for automatic corrections or perform manual calculations (SM 4500-O G).

3. PREVENTIVE MAINTENANCE: Refer to FT 1000, section 3.

4. DOCUMENTATION

4.1. Standard and Reagent Documentation: Document information about standards and reagents used for verifications.

4.1.1. Note the date of receipt, the expiration date and the date of first use for all standards and reagents.

4.1.1.1. Document acceptable verification of any standard used after its expiration date.

4.1.2. Record the concentration or other value for the standard in the appropriate measurement units.

4.1.2.1. Note vendor catalog number and description for pre-formulated solutions as well as for neat liquids and powdered standards.

4.1.2.2. Retain vendor assay specifications for standards as part of the calibration record.

4.1.3. Record the grade of standard or reagent used.

4.1.4. When formulated in-house, document all calculations used to formulate calibration standards.

4.1.4.1. Record the date of preparation for all in-house formulations.

4.1.5. Describe or cite the procedure(s) used to prepare any standards in-house (DEP SOP or internal SOP).

4.2. Field Instrument Calibration Documentation: Document acceptable calibration and calibration verification for each instrument unit and field test or analysis, linking this record with affected sample measurements.

4.2.1. Retain vendor certifications of all factory-calibrated instrumentation.

4.2.2. Designate the identity of specific instrumentation in the documentation with a unique description or code for each instrument unit used.

4.2.2.1. Record the manufacturer name, model number and identifying number such as a serial number for each instrument unit.

4.2.3. Record the time and date of all initial calibrations and all calibration verifications.

4.2.4. Record the instrument reading (value in appropriate measurement units) of all calibration verifications.

4.2.5. Record the temperature associated with all calibration verifications.

4.2.6. Record the name of the analyst(s) performing the calibration.

4.2.7. Document the specific standards used to calibrate or verify the instrument or field test with the following information:

- Type of standard or standard name (e.g., saturation)
- Value of standard, including correct units (e.g., mg/L at ^{o}C)
- Link to information recorded according to section 4.1 above

4.2.8. Retain manufacturers' instrument specifications.

4.2.9. Document whether successful initial calibration occurred.

4.2.10. Document whether each calibration verification passed or failed.

4.2.11. Document any corrective actions taken to correct instrument performance according to records requirements of FD 3000.

4.2.11.1. Document the date and time of any corrective action.

4.2.11.2. Note any incidence of discontinuation of use of the instrument due to calibration failure.

4.2.12. Describe or cite the specific calibration or verification procedure performed (DEP SOP or internal SOP).

4.3. Record all field-testing measurement data, to include the following:

- Project name
- Date and time of measurement or test (including time zone, if applicable)
- Source and location of the measurement or test sample (e.g., monitoring well identification number, outfall number, station number or other description)
- Latitude and longitude of sampling source location (if required)
- Analyte or parameter measured
- Measurement or test sample value
- Reporting units
- Initials or name of analyst performing the measurement
- Unique identification of the specific instrument unit(s) used for the test(s)

Appendix FT 1500
Tables, Figures and Forms

Table FT 1500-1 Solubility of Oxygen in Water

Table FT 1500-1: Solubility of Oxygen in Water at Atmospheric Pressure[1,2]			
Temperature °C	Oxygen Solubility mg/L	Temperature °C	Oxygen Solubility mg/L
0.0	14.621	26.0	8.113
1.0	14.216	27.0	7.968
2.0	13.829	28.0	7.827
3.0	13.460	29.0	7.691
4.0	13.107	30.0	7.559
5.0	12.770	31.0	7.430
6.0	12.447	32.0	7.305
7.0	12.139	33.0	7.183
8.0	11.843	34.0	7.065
9.0	11.559	35.0	6.950
10.0	11.288	36.0	6.837
11.0	11.027	37.0	6.727
12.0	10.777	38.0	6.620
13.0	10.537	39.0	6.515
14.0	10.306	40.0	6.412
15.0	10.084	41.0	6.312
16.0	9.870	42.0	6.213
17.0	9.665	43.0	6.116
18.0	9.467	44.0	6.021
19.0	9.276	45.0	5.927
20.0	9.092	46.0	5.835
21.0	8.915	47.0	5.744
22.0	8.743	48.0	5.654
23.0	8.578	49.0	5.565
24.0	8.418	50.0	5.477
25.0	8.263		

1. The table provides three decimal places to aid interpolation

2. Under equilibrium conditions, the partial pressure of oxygen in air-saturated water is equal to that of the oxygen in water-saturated

FT 1600. Field Measurement of Turbidity

Use in conjunction with:

- FT 1000 General Field Testing and Measurement
- FS 1000 General Sampling Procedures
- FD 1000 Documentation Procedures

1. INTRODUCTION: Turbidity measures the scattering effect that suspended solids have on the propagation of light through a body of water (surface or ground waters). The higher the effect (i.e., intensity of scattered light), the higher the turbidity value. Suspended and colloidal matter such as clay, silt, finely divided organic and inorganic matter, and plankton and other microscopic organisms cause turbidity in water.

This SOP describes the use of true nephelometric measurement using instruments meeting the specifications outlined in 2.1.

Exceptions to the requirements specified in 2.1 below include:

1.1. *In situ* probes with turbidity sensors used for screening purposes (e.g., groundwater purge stabilization measurements).

1.2. Non standard light sources, detectors or other turbidity measuring devices may be proposed for use in studies that entail comparison measurements (dredge and fill) or unattended deployment for monitoring purposes.

1.3. **Do not report results from "non standard" sensors or configurations for regulatory purposes such as permit compliance unless the Department has approved the use for the specific project.**

1.4. All "non standard" instrument must be calibrated/check according to the principles outlined in this SOP.

2. EQUIPMENT AND SUPPLIES

2.1. Field Instrument: Use a turbidimeter (nephelometer) or a spectrophotometer consisting of a light source and one or more photoelectric detectors with a readout device to indicate the intensity of light. The instrument must meet these specifications:

2.1.1. The light source must have a tungsten-filament lamp operated at a color temperature between 2000 and 3000 K.

2.1.2. The distance traversed by the incident light and scattered light within the sample tube must not exceed 10 cm.

2.1.3. The light detector, positioned at 90^{o} to the incident light, must have an acceptance angle that does not exceed $\pm$ 30^{o} from 90^{o}.

2.1.4. The detector and any filter system must have a spectral peak response between 400 and 600 nanometers.

2.1.5. The instrument sensitivity must permit detection of a turbidity difference of 0.02 NTU at the 0 – 1.0 NTU scale.

2.1.6. Note: using the appropriate equipment and following the procedures in this SOP, the field accuracy of this measurement is close to %R = 100 ± 10% for turbidities in the range of 1 to 100 NTU.

2.2. Sample Cells (cuvettes): Use sample cells or tubes of clear, colorless glass or plastic.

2.2.1. Keep cells clean, both inside and out, and discard if scratched or etched.

2.2.1.1. Never handle them where the light beam strikes the sample.

2.2.1.2. Clean sample cells by thorough washing with laboratory soap (inside and out) followed by multiple rinses with distilled or de-ionized water, and let air-dry.

2.2.2. Use a very thin layer of silicone oil on the outside surfaces to mask minor imperfections or scratches in the cells.

2.2.2.1. Use silicone oil with the same refractive index of the glass; making sure the cell appear to be nearly dry with little or no visible signs of oil.

2.2.3. Because small differences between cells significantly impact measurement, use either matched pairs or the same cell for standardization and sample measurement.

2.3. Standards:

2.3.1. Primary standards: Use these standards for initial calibration.

2.3.1.1. Formazin standards can be either obtained commercially or prepared according to method SM 2130B, section 3.b. See *Standard Methods for the Examination of Water and Wastewater* (American Public Health Association, American Water Works Association, Water Pollution Control Federation).

2.3.1.2. Some instruments may require the use of styrene divinylbenzene (SDVB) standards for calibration.

2.3.2. Secondary Standards: Use only those certified by the manufacturer for a specific instrument. Secondary standards must only be used for continuing calibration verifications according to the procedures in section 3.4 below. Determine or verify the values of secondary standards according to the procedure in section 3.3 below.

2.3.3. Turbidity-free water: Use filtered, laboratory reagent water demonstrated to be free of measurable turbidity (<0.01 NTU) or purchase commercially prepared turbidity-free water.

3. CALIBRATION AND USE

3.1. General Concerns

3.1.1. Light absorption by dissolved and suspended matter may cause a negative bias on the turbidity measurement. When present in significant concentrations, particles of light-absorbing materials such as activated carbon will cause a negative interference. Likewise, the presence of dissolved, color-causing substances that absorb light may also cause a negative interference. Some commercial instruments may have the capability of either correcting for slight color interference or optically blanking out the color effect.

3.1.2. Handle samples with natural effervescence as described in 3.5.5.1 below.

3.2. Calibration and Initial Calibration Verification

3.2.1. Follow the calibration activities in FT 1000, section 2.2.

3.2.2. Perform an initial calibration using at least two primary standards.

3.2.2.1. If the instrument cannot be calibrated with two standards, calibrate the instrument with one standard and verify with a second standard per 3.2.3 below.

3.2.2.2. For measurement of samples of very low turbidity, select the lowest standard commercially available for bracketing the lower end of the anticipated sample turbidity range or dilute higher turbidity standards with turbidity-free water.

3.2.2.3. Do not use turbidity-free water as a calibration verification standard.

3.2.3. Perform an initial calibration verification by reading at least one primary standard as a sample. The acceptance criterion for the initial calibration verification depends on the range of turbidity of the standard value:

- Standard Value = 0.1-10 NTU: the response must be within 10% of the standard;
- Standard Value = 11-40 NTU: the response must be within 8% of the standard;
- Standard Value = 41-100 NTU: the response must be within 6.5% of the standard; and
- Standard Value > 100 NTU: the response must be within 5% of the standard.

3.3. Determining the Values of Secondary Standards

3.3.1. Use only those standards certified by the manufacturer for a specific instrument.

3.3.2. Use verified secondary standards only for continuing calibration verifications.

3.3.3. Determining the initial value(s) of secondary standard(s):

3.3.3.1. Calibrate or verify the instrument with primary standards. Select primary standards that bracket the range of the secondary standards.

3.3.3.2. Immediately after the an initial calibration with primary standards or verification with a primary standard, read each secondary standard as a sample use the reading from the instrument as the first assigned value.

3.3.4. Verifying Secondary Standards

3.3.4.1. At least once per quarter or at other documented intervals (see 3.3.5 below), determine or verify the values of secondary standards immediately after the instrument has been calibrated or verified with primary standards.

3.3.4.2. Read each secondary standard as a sample. This reading must be within the manufacturer's stated tolerance range and within the acceptance ranges of the assigned standard value as listed in 3.2.3., above. If the criteria in section 3.2.3., above are not met, assign this reading as the value of the standard. If the reading is outside the manufacturer's stated tolerance range, discard the secondary standard.

3.3.5. **More frequent calibration verifications may be required for discharge permit compliance measurements or other regulatory requirements.**

3.4. Continuing Calibration Verification: Perform a continuing calibration verification using at least one primary or secondary standard. The calibration acceptance criteria are the same as those listed in section 3.2.3 above.

3.5. Measuring Turbidity in Samples

3.5.1. Gently agitate the sample and wait until air bubbles disappear.

3.5.2. Double-rinse the sample cell or cuvette with a small amount of the sample. Discard, and pour an aliquot into the sample cell or cuvette.

3.5.3. Gently dry out its external surface with lint-free paper.

3.5.4. Insert the cell in the instrument and read the turbidity directly from the meter display.

3.5.5. Do not use vacuum degassing, ultrasonic bath or other devices to remove bubbles from the sample. If the sample contains visible bubbles or if it effervesces (as in groundwater, with changes in pressure and temperature), make a note of this in the field records and collect a sample for laboratory measurement.

3.5.5.1. If effervescing samples are collected for laboratory analysis collect the sample without leaving headspace in the container and ship it as soon as possible to the laboratory (the holding time for this measurement is only 48 hrs). Ship this sample in wet ice at 4^{o}C.

3.5.6. Pour out the sample, double-rinse the cuvette with de-ionized water in preparation for the next sample.

4. PREVENTIVE MAINTENANCE: Refer to FT 1000, section 3.

5. DOCUMENTATION

5.1. Standard and Reagent Documentation: Document information about standards and reagents used for calibrations, verifications, and sample measurements.

5.1.1. Note the date of receipt, the expiration date and the date of first use for all standards and reagents.

5.1.1.1. Document acceptable verification of any standard used after its expiration date.

5.1.2. Record the concentration or other value for the standard in the appropriate measurement units.

5.1.2.1. Note vendor catalog number and description for preformulated solutions as well as for neat liquids and powdered standards.

5.1.2.2. Retain vendor assay specifications for standards as part of the calibration record.

5.1.3. Record the grade of standard or reagent used.

5.1.4. When formulated in-house, document all calculations used to formulate calibration standards.

5.1.4.1. Record the date of preparation for all in-house formulations.

5.1.5. Describe or cite the procedure(s) used to prepare any standards in-house (DEP SOP or internal SOP).

5.2. Field Instrument Calibration Documentation: Document acceptable calibration and calibration verification for each instrument unit and field test or analysis, linking this record with affected sample measurements.

5.2.1. Retain vendor certifications of all factory-calibrated instrumentation.

5.2.2. Designate the identity of specific instrumentation in the documentation with a unique description or code for each instrument unit used.

5.2.2.1. Record manufacturer name, model number, and identifying number (such as a serial number) for each instrument unit.

5.2.3. Record the time and date of all initial calibrations and all calibration verifications.

5.2.4. Record the instrument reading (value in appropriate measurement units) of all calibration verifications.

5.2.5. Record the name of the analyst(s) performing the calibration.

5.2.6. Document the specific standards used to calibrate or verify the instrument or field test with the following information:

- Type of standard or standard name (e.g., formazin)
- Value of standard, including correct units (e.g., 20 NTU)
- Link to information recorded according to section 5.1 above

5.2.7. Retain manufacturers' instrument specifications.

5.2.8. Document whether successful initial calibration occurred.

5.2.9. Document whether each calibration verification passed or failed.

5.2.10. Document any corrective actions taken to correct instrument performance according to records requirements of FD 3000.

5.2.10.1. Document date and time of any corrective action.

5.2.10.2. Note any incidence of discontinuation of use of the instrument due to calibration failure.

5.2.11. Describe or cite the specific calibration or verification procedure performed (DEP SOP or internal SOP).

5.3. Record all field-testing measurement data, to include the following:

- Project name
- Date and time of measurement or test (including time zone, if applicable)
- Source and location of the measurement or test sample (e.g., monitoring well identification number, outfall number, station number or other description)
- Latitude and longitude of sampling source location (if required)
- Analyte or parameter measured
- Measurement or test sample value
- Reporting units
- Initials or name of analyst performing the measurement
- Unique identification of the specific instrument unit(s) used for the test(s)

Standard Parts

The following figure provides the standard parts for the UVOST. Prior to using the UVOST, these parts or satisfactory substitutions should be on hand or readily available when needed.

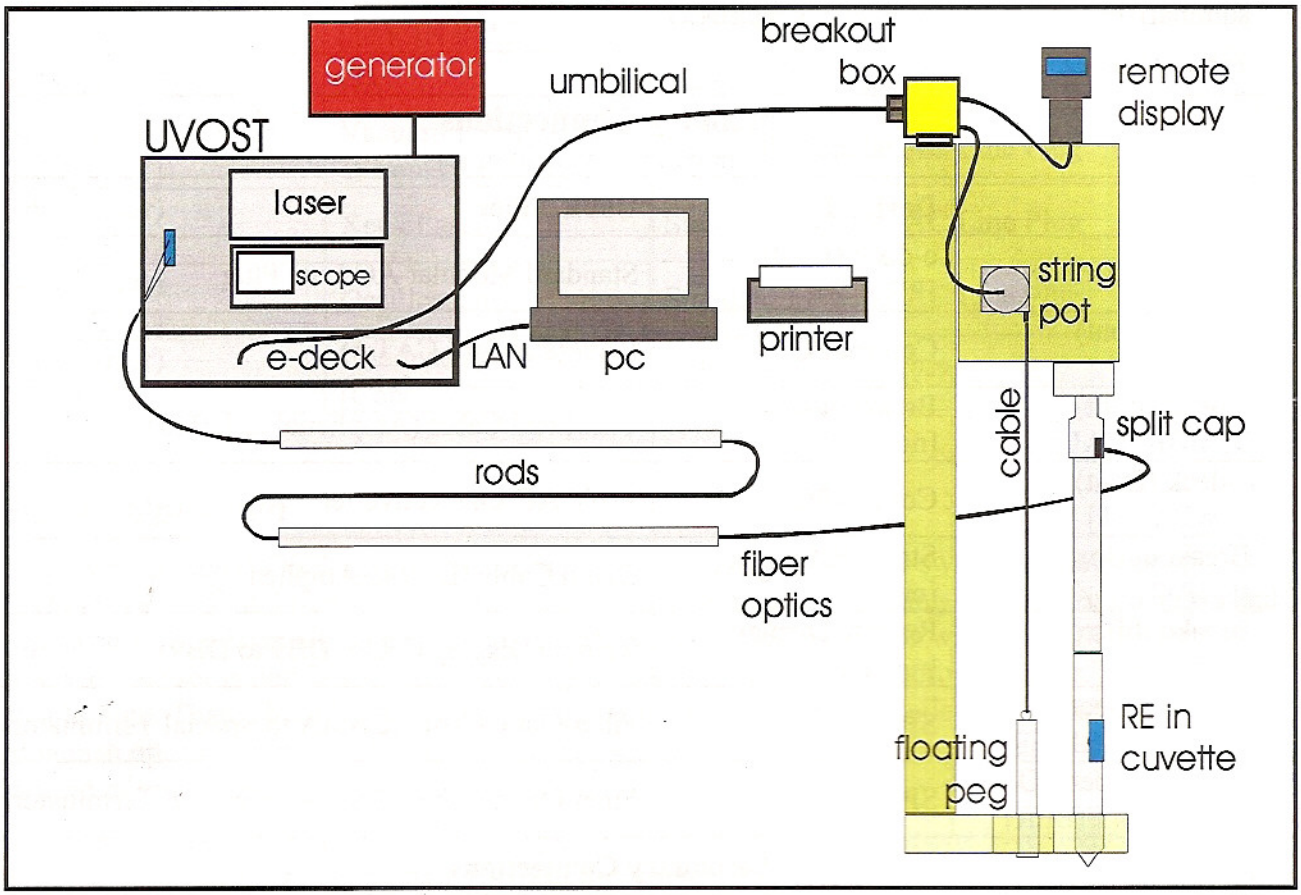


Figure 1. Standard UVOST parts.

General Operating Procedures

Set Up

Prior to operation, all the sub-systems require proper connections/cabling and power. Table 2 summarizes the proper connections/cabling.

Table 2. Cabling/Connections

Primary Connections [Connection labels in blue]		
Device 1	**Device 2**	**Cable/Fiber**
Power/Generator	e-deck (front) [PWR IN]	Standard Modular AC Line Plug
e-deck (front) [NET]	Control PC	LAN (standard CAT 5)
e-deck (front) [UMBILICAL]	Breakout Box [no label]	Umbilical Cable (Amphenol to DB15)
e-deck (front) [PWR OUT]	Control PC	110V AC Line converter
Breakout Box [DEPTH]	String Pot [no label]	Depth Cable (DB9 to Amphenol)
Breakout Box [DISLAY]	Remote Display [no label]	Remote Display Cable (DB9 to DB9)
UVOST Fiber I/O [LAUNCH FIBER]	SPOC	Fiber Optic Cable (2 SMA to Special Terminator)
UVOST Fiber I/O [RETURN FIBER]	SPOC	Fiber Optic Cable (2 SMA to Special Terminator)
Secondary Connections		
e-deck (front) [GPS]	GPS NMEA Output	DB9 RS-232 (Serial – usually integral to GPS)
e-deck (front) [AUX COM]	AD4 Quadrature	DB9 RS-232 (Serial)
e-deck (front) [12V AUX]	Generic 12V Accessory	Power Plug 0.1" (Switchcraft 761K)
Breakout Box [AUX]	NA (future use)	DB15 to (yet defined)
Permanent Connections		
Device 1	**Device 2**	**Cable/Fiber**
UVOST Fiber I/O (lower backside)	Detection Module (FIBER RETURN)	Single Fiber Optic (SMA-SMA)
UVOST Fiber I/O (upper backside)	Laser Launch Optics	Single Fiber Optic (High Power SMA-SMA) (standard fiber can be used as backup)
Trigger Photodiode	Oscilloscope [Ch1]	Coaxial Cable (SMA-BNC)
Emission Module [SIGNAL OUT]	Oscilloscope [Ch2]	Shielded Coax (PMT-BNC)

Trigger Photodiode	XeCl Laser (vessel)	Single Fiber Optic (SMA-SMA)
UVOST e-deck (rear) [NET SCP]	Oscilloscope	LAN (CrossOver CAT 5)
UVOST e-deck (rear) [12V PMT]	Detection Module [12V IN PMT]	12V supply (SMA-SMA)
AC Line (external)	NA	Standard Modular AC Line Plug
e-deck (rear left-most) [PWR OUT]	Vacuum Pump (switch at front of e-deck)	Standard Modular AC Line Plug
e-deck (rear) [PWR OUT]	XeCl Laser	Standard Modular AC Line Plug
e-deck (rear) [PWR OUT]	Oscilloscope	Standard Modular AC Line Plug
e-deck (rear) [Cond.]	Conductivity Module [Cond. Out]	12V Signal Cable (?-?)

Power Up/Down

To power up the UVOST, simply switch the power on using the power switch on the front of UVOST's e-deck. All peripheral devices are powered through the cabling – minimizing tangles and trip hazards. The laser takes several minutes of warm-up and Wait LED will then light. Once warmed the user is notified by the Ready light. Push the On button to activate lasing. Lasing LED should light. There should be a small rectangular yellow glow on the yellow glass indicator at front of launch optics assembly. The oscilloscope should display Trig'd – if laser has sufficient output (not in need of recharge).

Set laser rep rate to between 63-65 Hz. If powering up from cold conditions (overnight, etc.), make sure you have laser running at least 10-15 minutes prior to attempting your first RE calibration. We recommend running heaters overnight if in sub-freezing conditions to minimize warm-up times in the morning. Extremely high or low temperatures negatively affect laser power. If used in extreme conditions one should attempt to house/store the UVOST system in a warmer/cooler environment to assure proper operation. There are no hard/fast rules for this – since case temperatures/heaters can assist but a lot depends on winds, ventilation, direct sun, etc.

To power down the UVOST, first Stop the laser pulsing, then switch off the power button.

Boot PC and Check Software Function

Make sure all drivers are loaded and ready. Start the OST system software. Indicators in the software will assist in alerting you to problem connections and general status of the components (Hardware Tab). See software manual for specifics on OST software.

Proper System Function

Once the software is started and functional you can proceed to check the depth encoding and associated peripheral functions. Actuating the probe (or hand advancing the string pot) should show Current Depth changing on the OST software (Depth tab). The Remote Display should be functional and show status. Activate Info tab and make sure your job information is updated for storage with each LIF log.

SPOC Setup

A detailed discussion is available under SPOC Assembly heading. Carefully examine mirror and window for ANY trace grease, lint, and moisture. They must be very clean. Assure that all o-rings, seals, and adapters are in correct order – including Teflon tape, and associated hardware. With SPOC tip left off of SPOC, dry the air inside the SPOC, and quickly screw in window. You can check for moisture condensing inside window using an ice cube. If there is condensation you must dry the SPOC air better. Slightly tighten the mirror and fiber optic Swagelock seals (just snug). Adjust fiber terminator up/down to achieve proper distance from mirror to collimate the laser beam (use white paper – you may have to "up" energy for this).

Place RE in front of window and adjust laser energy (Fiber I/O block screw) to achieve approximately ¾ scale with oscilloscope's CH2 on 50 mV/div. Adjust the mirror (using window pick/hook) to image only the sapphire window – not epoxy or SPOC barrel (no clipping – full circle image on paper). This occurs approximately 1/3 of the way down from top of window.

Clean/polish window and then make sure that background does not exceed ~2.5mV peak signals. If background is high, carefully inspect for imaging of sides/epoxy or contamination (lint, cotton fibers, fuel, moisture, grease, etc.) An unacceptably high background can make interpretation extremely difficult.

Once you're certain the mirror/fiber/window system is achieving proper results you can tighten the Swagelocks securely. Use ONLY the supplied wrenches to hold the SPOC securely during tightening. This is most readily assured by laying SPOC down and only handling wrenches. Use the mirror pick/hook to hold the mirror firmly in place during tightening to prevent rotation. Make sure laser beam stays in centered in the window (side to side) and 1/3 down from the top (toward first rod).

With window/mirror/fiber terminator all secured, proceed with attaching drive tip, adapter, extension rod, and tighten extremely well with 2 pipe wrenches or pipe wrench and vice. Teflon tape helps reduce loosening from rattling/vibration.

Background

Wipe window clean and acquire a Background (blank) waveform with the Acq BckG command. A perfect system would yield no waveform at all – only white noise. But there is always trace fluorescence from mirror/window, fiber-generated Raman, and contamination. Try to achieve <2mV peak signal in any one channel. You simply want it as small as you can get it. A severely jagged/noisy background indicates possible pickup of the large laser EMF (Electric and Magnetic Fields) into the trigger and signal coax cables. Loose grounds, connections, misrouting of cables, etc. can induce this. If the first channel (350 nm) is considerably large than the other three, there is a chance that you have excessive backscatter of laser light into the system (350nm filter is near laser wavelength) or the laser rejection filter (inside I/O block) may be damaged or malfunctioning. Channels 3 and 4 being high/narrow is a classic lint signature. A background waveform that looks like your current contaminant of interest suggests leakage and contamination of the internal SPOC mirror/window OR simply a dirty window. Clean with methanol or solvent if soap/water doesn't work.

RE Calibration

Calibration should be done as immediately preceding each UVOST logging event. Don't calibrate with RE, then spend time monkeying around with push rig, etc. Wait until the direct push rig is ready to go. Pre-push with dummy tip if obstructions are likely or getting a "straight hole going" is

difficult. Place RE holder on window (making sure window is very clean). Immediately acquire RE with Acq RE command. Extended exposure to laser light can form excimers and photodegradation – causing a morph in waveform shape/intensity. If you have changed fiber optic lengths the software may correct the delay time to achieve proper position in window. Make sure the RE signal level exceeds a 10, 000 pVs minimum but does not exceed 20,000 pVs with 14,000-15,000 pVs about optimum. Try to be consistent (± 1000 pVs) – especially when on the same project/site. Make sure the RE waveform shape "looks right". Compare it to the reference waveform displayed on the scope during the RE acquisition. Extremely noisy/jagged REs, misshapen REs, and missing/low channel contributions indicate damaged or loose fiber optics/filters/detector.

Logging

Follow these steps to acquire a UVOST log:

Step 1. With proper RE and background acquired, pertinent log information recorded, and probe in position (window just below (~1 inch) ground surface), activate the Record command.

Step 2. If you failed to acquire a recent RE the OST software will alert you that it's not recent (at least one log event old). Proceed with you recent (perhaps you just aborted a "false start"/crooked log) – or cancel out and acquire the RE you forgot to acquire. You can "rescue" an RE if it's for a rational purpose (such as an accidentally aborted log and you want to continue logging and probe is under ground, under water during a barge project, etc.) DO NOT purposefully continue logging without a new RE for each and every log if you're having problems acquiring a new RE due to a problem. FIX the problem, acquire a good RE, then proceed. Failure to acquire a new RE for each log will generate inaccurate data.

Step 3. Choose a directory and name for your log. UVOST auto-suggests the name sequentially in an attempt to reduce typing. In order to absolutely avoid accidental overwrite of any OST file, the OST software creates a unique time/date name and uses that name in place of overwrites (even though you said "OK" to the overwrite. If you want to risk it, you can always delete a file from the Save File dialog after you click on it once, but before hitting OK. That prevents the Windows software from reporting an overwrite to the OST and cueing the unique filename routine. The safest method is to choose OK to overwrite – and rename files later.

Step 4. Once the name is chosen you are asked to choose whether or not to "zero" the depth. For normal logs you always choose Yes and zero out depth. If you're continuing an aborted log that you want to continue (accidental termination) – choose No. Log should continue at depth where you left off.

Step 5. As the log progresses, it is your responsibility to make sure the system is operating properly. Observe the oscilloscope or OST display to watch for unusual events such as:

A. Try to keep the probe advancing at approximately 0.75 inch/sec – your company may choose less – but we do not recommend faster

B. Strange background drifts several feet under (possible fogging), etc.

C. Broken depth cable or poor connection will result in jumps in depth or a loss of depth increase – even though the operator is advancing the probe

D. Incorrect depths would indicate a possible rod length or string pot cal factor mismatch

E. Sudden loss of waveform (flatline) indicates possible fiber optic break due to broken probe

F. depth is advancing – but no new waveform updates aren't showing up – this indicates poor triggering – is Trig'd showing up on oscilloscope every second or so? if not – hit Trigger

50% button on scope or look for other cause such as Stop button on laser being accidentally pushed.

Step 6. Once refusal is reached – or target depth is reached – activate the End command. All pertinent data is stored and the oscilloscope scale is automatically returned to the default 50mV/div scale in preparation for next RE.

Step 7. Inspect the probe, window, etc. for leaks, breaks, and loose parts in preparation for next the next logging event (push).

Printing/Exporting LIF Logs

Once the push is complete the log can be viewed (a log can be also opened from file and viewed with the OST software) it is necessary to print the log to paper or export it to an electronic image (JPG file). Prior to print/export it is most often desirable to select callout waveforms. Select single waveforms by clicking the log at any depth – which creates a stats bar. Transfer single logs by dragging/dropping the stats bar or with the < bar next to each callout box. Select the average of a region of waveforms along a log by clicking the log, holding down, then releasing at a second depth along the log. Transfer average zone waveforms by dragging/dropping the bottom stats bar or with the < bar next to each callout box. Reasons to select certain depths/regions include:

- Bracketing what appear to be continually affected zones - this helps the client/consultant "summarize" the general NAPL zones and easily jot down depths for future validation sampling, project design, discussion with site owner, etc.
- It's best to bracket large zones of homogenous NAPL - do not span different products
- Highlighting unusual signatures – perhaps to suggest sampling there or to "flag" things the client needs to investigate or discount
- Maybe a background here/there to remind viewer what "clean" looks like
- Any potential "false positives" such as mineral/plant/urban background/highly degraded NAPL – the different waveform should help client understand that "it's nothing to worry about"
- Use caution when highlighting single waveforms from the rising edge of NAPL hits – the waveforms in these area are usually saturated because the oscilloscope scaling wasn't able to fully respond – they are morphed and ugly and cause unnecessary confusion and alarm
- You do not have to start with top and work down – pick a callout "straight across" for neater appearance
- Avoid "crossing" of the depths of multiple callouts as this looks messy/confusing

It is best that the UVOST operator and the client discuss depth/RE scales, depths of interest, etc. ahead of time to hopefully avoid lots of "reprints".

It is suggested that you annotate the callouts (text box under each waveform) in order to guide the client. If it's the usual product you expect then leave it blank – but if it's unusual, significant, or out of the ordinary, guide the viewer with a brief description.

Each time you print/export the settings are saved in a lif.plt (plot) file. That way the same callouts and depths are available later. The OST software (and we) suggest that the very first print/export a log in the filed you save it as field. That way you always know what the client received originally. Subsequent print schemes are saved as well. Later, upon opening, you can choose which of the various schemes to open the file with.

Dakota Technologies
UVOST Log Reference

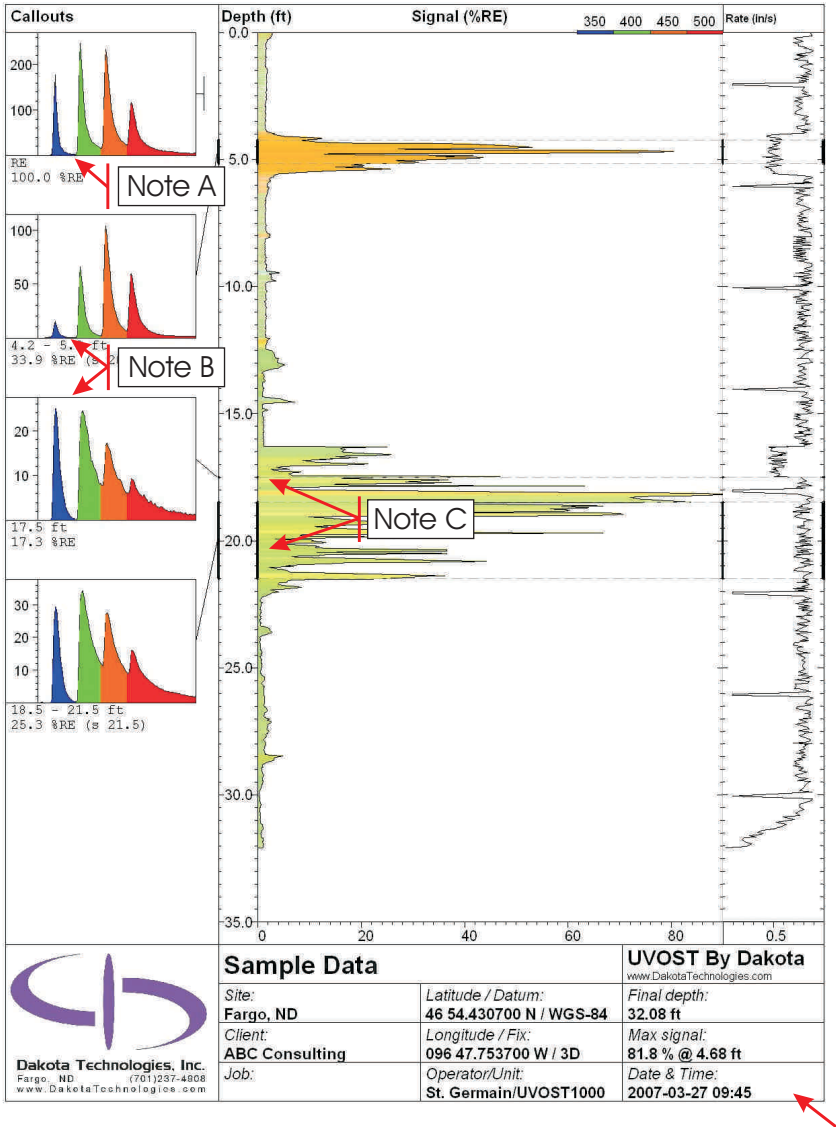
Main Plot :
Signal (total fluorescence) versus depth where signal is relative to the Reference Emitter (RE). The total area of the waveform is divided by the total area of the Reference Emitter yielding the %RE. This %RE scales with the NAPL fluorescence. The fill color is based on relative contribution of each channel's area to the total waveform area (see callout waveform). The channel-to-color relationship and corresponding wavelengths are given in the upper right corner of the main plot.

Callouts :
Waveforms from selected depths or depth ranges showing the multi-wavelength waveform for that depth.

The four peaks are due to fluorescence at four wavelengths and referred to as “channels”. Each channel is assigned a color.

Various NAPLs will have a unique waveform "fingerprint" due to the relative amplitude of the four channels and/or broadening of one or more channels.

Basic waveform statistics and any operator notes are given below the callout.



Rate Plot :
The rate of probe advancement. Less than 0.8in (2cm) per second is preferred. A noticeable decrease in the rate of advancement may be indicative of difficult probing conditions (gravel, angular sands, etc.).

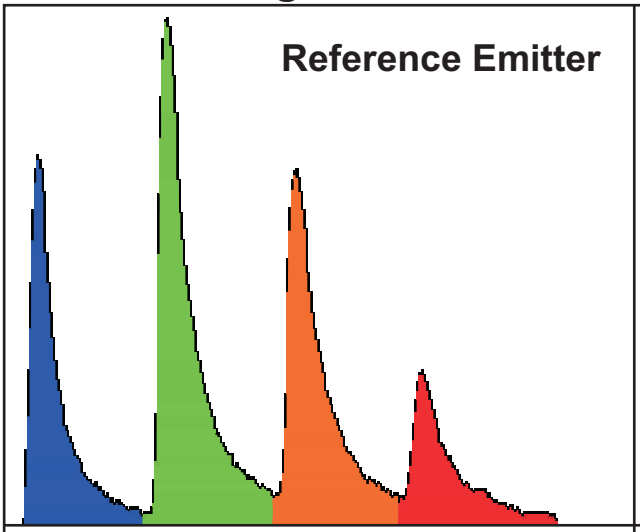
Info Box :
Contains pertinent log info including name and location.

Note A :
Time is along the x axis. No scale is given, but it is constant and is roughly 300ns wide.
The y axis is in mV and directly corresponds to the amount of light striking the photodetector.

Note B :
These two waveforms show two different products, each with a unique waveform. The first is used motor oil and the second is diesel.

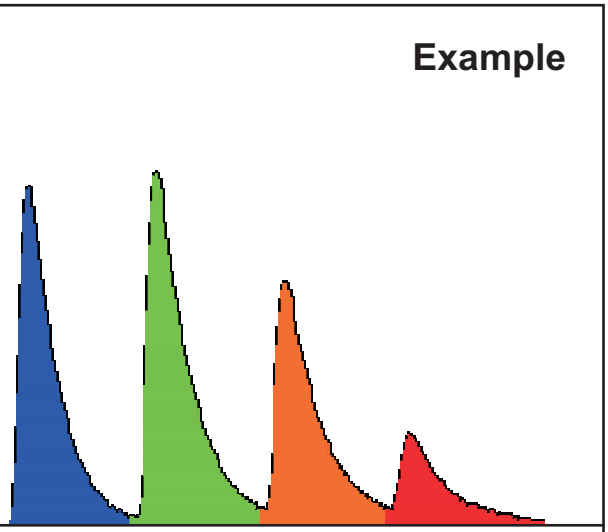
Note C :
Callouts can be a single depth (see 3rd callout) or a range (see 4th callout). The range is noted on the depth axis by a bold line. When the callout is a range, the average and standard deviation in %RE is given below the callout.

Waveform Signal Calculation



CH1		CH2		CH3		CH4		Total
4820	+	8108	+	6249	+	2984	=	**22161**
21.7		36.6		28.2		13.5		**100%**

Channel Area (pVs) Percent RE

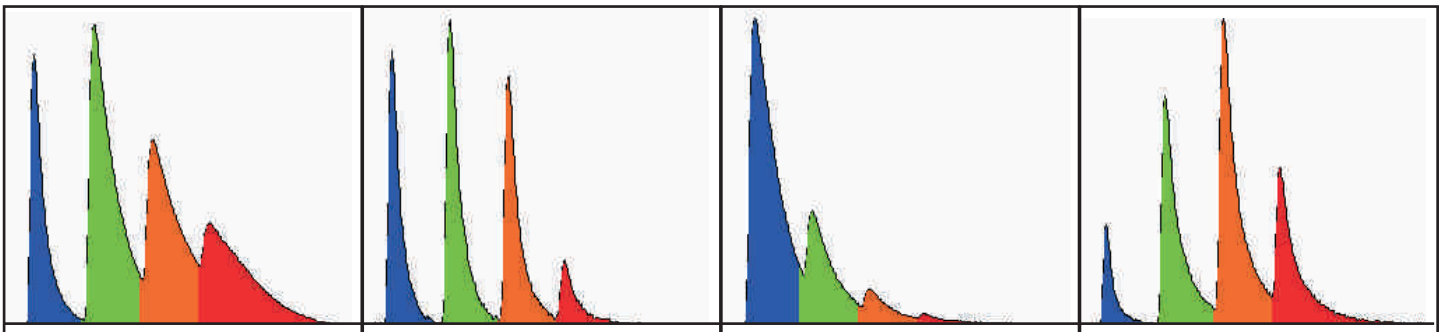


CH1		CH2		CH3		CH4		Total
4923	+	5743	+	4166	+	1735	=	**16587**
22.3		25.9		18.8		7.8		**75%**

Data Files

***.lif.raw.bin**	Raw data file. Header is ASCII format and contains information stored when the file was initially written (e.g. date, total depth, max signal, gps, etc., and any information entered by the operator). All raw waveforms are appended to the bottom of the file in a binary format.
***.lif.plt**	Stores the plot scheme history (e.g. callout depths) for associated Raw file. Transfer along with the Raw file in order to recall previous plots.
***.lif.jpg**	A jpg image of the OST log including the main signal vs. depth plot, callouts, information, etc.
***.lif.dat.txt**	Data export of a single Raw file. ASCII tab delimited format. No string header is provided for the columns (to make importing into other programs easier). Each row is a unique depth reading. The columns are: Depth, Total Signal (%RE), Ch1%, Ch2%, Ch3%, Ch4%. Summing channels 1 to 4 yields the Total Signal.
***.lif.sum.txt**	A summary file for a number of Raw files. ASCII tab delimited format. The file contains a string header. The summary includes one row for each Raw file and contains information for each file including: the file name, gps coordinates, max depth, max signal, and depth at which the max signal occured.
***.lif.log.txt**	An activity log generated automatically located in the OST application directory in the 'log' subfolder. Each OST unit the computer operates will generate a separate log file per month. A log file contains much of the header information contained within each separate Raw file, including: date, total depth, max signal, etc.

Common Waveforms (highly dependent on soil, weathering, etc.)



Diesel	Gas	Kerosene	Motor Oil

JOB SAFETY ANALYSIS **Columbia Technologies**	DATE 6/30/2008 Revised: 7/27/2010 By: A. Heckhaus	☐ NEW ☒ REVISED	

JSA TYPE CATEGORY	WORK TYPE	WORK ACTIVITY (Description)	
Active and Inactive Retail and terminal locations and other applicable work locations	**Assessment and Remediation**	**Membrane Interface Probe (MIP) Operation**	
DEVELOPMENT TEAM	**POSITION / TITLE**	**REVIEWED BY:**	**POSITION / TITLE**
Doug McInnes	MIP Operator	John H. Sohl	CEO

REQUIRED PERSONAL PROTECTIVE EQUIPMENT

☒ HARD HAT; ☒ SAFETY GLASSES; ☒ HEARING PROTECTION
☒ SAFETY SHOES Steel Toes & non slip soles; ☒ PPE CLOTHING Orange coveralls or safety vest
☒ GLOVES Leather, Nitrile
☒ OTHER Respirators may be required if conditions or work area air quality exceeds applicable HASP Action Levels

REQUIRED AND/OR RECOMMENDED TOOLS AND EQUIPMENT

JOB STEPS	POTENTIAL HAZARDS	CRITICAL ACTIONS
Review "General Site Activities" JSA		
Initialize MIP System		
Setup MIP system for operation	Electrocution/Contact with energized electrical lines	• Inspect instrument power cords each day, replace or repair if worn • During rain events, protect energized equipment by closing access doors, or provide moisture barrier • All circuits must include a GFI safety device
	Caught between moving parts when handling heavy objects	• Provide and wear proper work gloves when the possibility of crush, pinch, or other injury may be caused by moving/stationary edges or objects.
	Injury from compressed gasses	• Transport compressed gas cylinders properly secured • During mobilization, secure compressed gas cylinders with safety caps
	Fire/Explosion	• Obtain hot work permit, if required, for generator or FID (hydrogen flame) use • Prohibit smoking in the MIP unit • Provide ABC (or equivalent) fire extinguisher • Store flammable liquids in well ventilated areas. • Prohibit storage and transfer of flammable liquids in plastic containers. • Store combustible materials away from flammables.
Enable temperature block heating	Exposure to High Temperatures	• The MIP probe is operated at a temperature of 121 C, about 250 F, which can cause damage to plastic objects and can cause burns to exposed skin • Wear heat resistant leather gloves with a maximum temperature rating of at least 121° C (250°F) when handling a heated MIP probe • Make note of the heated zone of the MIP probe during initial safety meetings, and daily tailgate meetings when workers or visitors not familiar with MIP operations are present onsite. • Do not allow the heated surface of the MIP probe to come into contact with plastic/paper/easily ignited materials. Always store the heated MIP probe on the metal rod rack, or on a non-flammable surface when above ground surface

Enable gas flow	Contact with Performance Test Reagents	• Wear nitrile or latex gloves when preparing performance test stock solutions, or when preparing performance test aqueous solutions • Wear nitrile or latex gloves when immersing the MIP probe in performance test solutions • Properly containerize and dispose of performance test solutions • Avoid breathing any steam or vapor produced during performance tests • Provide air montiroing to employees during performance test. • Provide workers proper skin, eye, and respiratory protection based on the exposure hazards present. • Maintain MSDS sheets for all hazardous substances brought onsite.

JOB SAFETY ANALYSIS **Columbia Technologies**	DATE 2/27/2009 Evaluated: 1/1/2010 By: A. Heckhaus	☒ NEW ☐ REVISED	

JSA TYPE CATEGORY	WORK TYPE	WORK ACTIVITY (Description)	
Active and Inactive Retail and terminal locations and other applicable work locations	**Assessment and Remediation**	**Laser Induced Fluorescence (LIF) Operation**	
DEVELOPMENT TEAM	**POSITION / TITLE**	**REVIEWED BY:**	**POSITION / TITLE**
Amy Heckhaus	LIF Operator	Kevin VanDeVusse	Operations Manager

REQUIRED PERSONAL PROTECTIVE EQUIPMENT

☒ HARD HAT; ☒ SAFETY GLASSES; ☒ HEARING PROTECTION
☒ SAFETY SHOES Steel Toes & non slip soles; ☒ PPE CLOTHING Orange coveralls or safety vest
☒ GLOVES Leather, Nitrile
☒ OTHER Respirators may be required if conditions or work area air quality exceeds applicable HASP Action Levels

REQUIRED AND/OR RECOMMENDED TOOLS AND EQUIPMENT

JOB STEPS	POTENTIAL HAZARDS	CRITICAL ACTIONS
Review "General Site Activities" JSA		
Initialize LIF System		
Setup LIF system for operation	Electrocution/Contact with energized electrical lines Caught between moving parts when handling heavy objects/Pinch Points Fire/Explosion	• Inspect instrument power cords each day, replace or repair if worn. • During rain events, protect energized equipment by closing access doors, or provide moisture barrier. • All circuits must include a GFI safety device. • Provide and wear proper work gloves when the possibility of crush, pinch, or other injury may be caused by moving/stationary edges or objects. • Obtain hot work permit, if required, for generator use. • Prohibit smoking around the LIF unit. • Provide ABC (or equivalent) fire extinguisher.
LIF Operation	Exposure to Laser/Sapphire Window Reference Emitter Chemical exposure	• Limit direct visual contact with laser/sapphire window. • Handle reference emitter carefully to ensure breakage of glass does not occur. • Maintain MSDS sheets for all hazardous substances brought onsite, and for anticipated subsurface contamination constituents.

Push Rig Operation	Caught between moving parts when handling heavy objects/Pinch Points Loud Noises Slip/Trip/Fall Injury during Movement of Vehicle/Equipment	• Provide and wear proper work gloves when the possibility of crush, pinch, or other injury may be caused by moving/stationary edges or objects. • Provide and wear proper hearing protection (ear muffs or ear plugs) when operating or in the vicinity of the push rig. • Survey boring location area prior to setup. • Maintain good housekeeping practices around equipment. • Prior to moving the vehicle/push equipment, perform a walk-around of equipment to ensure no hidden dangers are present. • If visual sight is limited, use a spotter to assist in moving equipment. • Maintain safe speed for the area. • Maintain safe distance from moving equipment.
Extracting rods from ground	Exposure to contaminated subsurface and/or free product	• Provide and wear proper hand protection (i.e. nitrile gloves) when extracting push rods from the ground. • If extremely saturated conditions are present protect face and body with appropriate safety measures (i.e. faceshield and disposable clothing, such as Tyvek.)

JOB SAFETY ANALYSIS **Columbia Technologies**	DATE 6/30/2009	☐ NEW ☒ REVISED	

JSA TYPE CATEGORY	WORK TYPE	WORK ACTIVITY (Description)
Active and Inactive Retail and terminal locations and other applicable work locations	**Assessment and Remediation**	**Geoprobing/Soil Sampling**

DEVELOPMENT TEAM	POSITION / TITLE	REVIEWED BY:	POSITION / TITLE
Amy Heckhaus	Operator	John H. Sohl	CEO

REQUIRED PERSONAL PROTECTIVE EQUIPMENT

☒ HARD HAT; ☒ SAFETY GLASSES; ☒ HEARING PROTECTION
☒ SAFETY SHOES Steel Toes & non slip soles; ☒ PPE CLOTHING Orange coveralls or safety vest
☒ GLOVES Leather, Nitrile
☒ OTHER Respirators may be required if conditions or work area air quality exceeds applicable HASP Action Levels

REQUIRED AND/OR RECOMMENDED TOOLS AND EQUIPMENT

JOB STEPS	POTENTIAL HAZARDS	CRITICAL ACTIONS
Review "General Site Activities" JSA		
Lockout/Tagout		
Lockout all possible electrical lines on-site	Electrocution/Contact with energized electrical lines	• Identify all utilities around the site before work commences. Contact local one-call center at least 3 days prior to work commencement. Conduct private utility clearance if necessary. • Cease work immediately if unknown utility markers are uncovered. • Utility clearance shall conform with 29 CFR 1926.955 (high voltage >700 kv): 15 feet phase-to-ground clearance; 31 feet phase-to-phase clearance.

Mobilize to sampling location		
Geoprobe set up Set up decon area	Struck by/Against Heavy Equipment, Flying Debris, Protruding Objects	• Wear reflective warning vests when exposed to vehicular traffic. • Isolate equipment swing areas. • Make eye contact with operators before approaching equipment. • Barricade or enclose the drilling area. • Require backup alarms on all heavy equipment. • Restrict work area entry to authorized personnel during drilling activities. • Wear hard hats, safety glasses with side shields or splash/face shields and goggles, and steel-toed safety boots at all times. • Understand and review hand signals.
	Contact with high pressure water	• Set up decon area/pad for augers (if decon on site), do not spray your face/body with water. Use proper PPE. • If using a hot water pressure washer be aware of high temperature water.
Open/cut concrete/asphalt or soil at sampling location	Injury from Sharp Objects	• Wear cut resistant work gloves when the possibility of lacerations or other injury may be caused by sharp edges or objects. • Maintain all tools in a safe condition. • Keep guards in place during use. • Observe work area and location of other personnel before lifting or moving objects with sharp edges.
Inspect Geoprobe Rig	Injury from Contact with obstructions	• Daily equipment inspections as per manufacturers' requirements. • Inspect Geoprobe® drill rig • Inspection of all emergency equipment (fire extinguishers, first aid kits, and eye washes). • Check well locations for underground and overhead utilities.
	Injury from Vehicle instability	• Observe mast set up, so no contact with overhead obstacles. • The geoprobe must be leveled prior to beginning advancement activities.
	Injury from equipment Pinch points	• Remind everyone never to leave hand tools on rig. The rig can move and cause their hand/arm/tools to become pinched.
Soil Sampling		
Hand clear soil boring location, if required	Damage to underground improvements	• Hand clear slowly, do not force through soil. You may contact/break underground lines ("soft dig" technologies are strongly recommended).
	Bodily injury	• Wear proper PPE: (safety glasses, hard hat, level D clothing, hard hat, and work gloves) during hand clearing activities.
	Muscle/joint strain	• Keep your feet shoulder-width apart to improve stability and to avoid back, neck and wrist strain injuries.

Begin Drilling, Soil Boring	Injury from rotating parts Contact to contaminants and Inhalation of organic vapors	• Stay an arms length away from moving/rotating parts (i.e. augers, drill drive shaft). • Use nitrile gloves while handling soil samples. Scan with a PID. • Monitor the air in the work area with a PID for elevated vapors during the drilling activities. • Discontinue activities if the PID reads >5 ppm sustained for 10 minutes.
Collect soil samples and/or install temporary pipe	Housekeeping Improper drum storage Slip on ice formations Cuts to the skin	• Stay alert while logging soil samples for all drilling activities. • Maintain your work area, keep walkways clean, tools picked up, and soil and wastewater in drums and should be placed out of walkways and traffic areas. • All drums must be labeled Non-hazardous with generator information. • If ice forms while drilling in colder months, apply sand or salt to the work area to increase traction. • If a sample jar breaks, wear leather work gloves during the cleanup to prevent cuts to the skin.
Preparation to mob to next drilling location or leave site.	Contact with overhead obstructions and equipment	• Insure an observer is watching the lowering of the drill rig mast so no lines or overhead obstacles are contacted. • Prior to lowering rig off of outriggers make sure all tools and personnel are clear of the drill rig. • Prior to driving to next location make sure auger racks are secured and that drilling mast is completed lowered. • **Note:** While rig is moving on site have spotters verify clearance so no overhead obstacles are contacted and no obstacles are hit while backing.

JOB SAFETY ANALYSIS **Columbia Technologies**	DATE 6/30/2008 Evaluated: 1/1/2010 By: A. Heckhaus	☐ NEW ☒ REVISED	

JSA TYPE CATEGORY	WORK TYPE	WORK ACTIVITY (Description)	
General Sites	**Assessment and Remediation**	**General Site Activities (Health & Safety Contingency Plan)**	
DEVELOPMENT TEAM	**POSITION / TITLE**	**REVIEWED BY:**	**POSITION / TITLE**
Ned Tillman	President, P.G.	John Sohl	CEO

REQUIRED PERSONAL PROTECTIVE EQUIPMENT	**RECOMMENDED PERSONAL PROTECTIVE EQUIPMENT**	
☒ SAFETY GLASSES ☒ PPE CLOTHING reflective safety vest or orange outerwear. ☒ GLOVES Nitrile exam gloves, leather gloves ☒ SAFETY SHOES Steel Toes ☒ LONG SLEEVE SHIRT	☒ FACE SHIELD ☒ AIR PURIFYING RESPIRATOR (half mask respirator)	☒ GOGGLES ☒ HEARING PROTECTION ☒ HARD HAT

REQUIRED AND/OR RECOMMENDED TOOLS AND EQUIPMENT			

JOB STEPS	**POTENTIAL HAZARDS**	**CRITICAL ACTIONS**
Complete JSA/H&SP/Safety Checklist/Tailgate Meeting	Parking vehicles, traffic flow/control, access difficulties	• Park in a secure area where vehicle is out of traffic pattern so that tailgate safety meeting can be performed safely.
Establish/Set up site control (traffic control)	Contact with vehicles	• Wear highly visible clothing such as orange reflective traffic vests or clothing. • Utilize cones/barricades/safety fence to establish work zones as indicated in the “Traffic Control Program” posted in the HASP. • Cone/Flag height must be at least 50” tall.
	Pedestrian contact	• Establish access points in the work zone to keep pedestrians and unintentional traffic out. • Inform facility personnel of work (restricted) area and do not permit unauthorized individuals (those not properly trained or wearing appropriate PPE) access to the exclusion zone. A spotter must be utilized when vehicles, including construction vehicles, are backing up to ensure a safe pathway.
	Backing, moving vehicles	• Use a spotter when available. If there is no spotter, the driver must get out and walk around the vehicle prior to backing/moving. • Look up to ensure that overhead wires or structures can be safely cleared. • Look down to identify unusual depressions, holes or debris that may interfere with backing/moving. • Observe fixed objects or parked, unoccupied vehicles. • Back slowly using rear view/side view mirrors frequently. • If backing vision is obscured, stop the vehicle every few feet to exit and recheck the backing route. • Remain constantly alert at all times while backing a vehicle for the potential for other vehicles or pedestrians to appear unexpectedly in the path of travel. • Vehicle tailgate must be in up/closed position when vehicle is in motion.
Remove/load equipment from vehicle	Back strain	• Utilize proper lifting procedure (keep your back straight) when loading coolers and/or equipment back into truck. (to avoid lifting heavy/awkward coolers leave cooler on tailgate to load samples and ice into). • Bend down at the knees and lift with your legs rather than bending and lifting with your back. • Utilize material handling devices when possible to move equipment (i.e. lift gates, pallet jacks, dollies, etc.) • If necessary, utilize a ramp for loading and unloading wheeled devices, ensuring the ramp is properly supported prior to use.

JOB SAFETY ANALYSIS **Columbia Technologies**	DATE 6/30/2008 Evaluated: 1/1/2010 By: A. Heckhaus	☐ NEW ☒ REVISED	PAGE 2 of 2

Monitor weather conditions	Weather related issues; cold/hear related injuries	• Monitor weather forecast. • Shut down operations should severe weather conditions exist, including lightning and excessive wind speed. • At the first sign of lightning, field operations will be suspended for 30 minutes after the last lightning strike. • Monitor for heat/cold stress • Provide fluids to prevent worker dehydration. • Staff should understand and be able to recognize the signs and/or symptoms of cold and hot weather related illnesses. • Personnel should dress appropriately for ambient temperatures that would include but not limited to dry layered clothing. • For cold weather, work schedules may be adjusted to provide sufficient break periods in a heated area and time intervals for replenishing fluids. • For hot weather, work schedules may be adjusted to provide sufficient break periods in an air conditioned area and time intervals for replenishing fluids.
	Exposure to site contaminants	• Review and understand Action Levels noted in the HASP. • Monitor (evaluate) breathing zone of workers with PID. • Monitor (evaluate) any enclosure with a PID. • Ensure that Level C PPE is available for potential upgrade.
Monitor Biological Conditions and Environment	Exposure to poisons and/or toxins	• Wear appropriate PPE (eye protection, long pants, Nitrile sampling gloves, shirt with sleeves, steel-toed shoes)
	Poisonous plants	• Do not touch or contact poisonous plants, such as poison ivy and poison oak. • If available, apply an over-the-counter barrier cream, such as Ivy Block® to prevent contact with plant oils. • Wash hands and arms immediately with soap and water if skin contacts the plants. • Wear long pants with socks pulled over legs to prevent skin contact with plants and insects. • Identify and review poisonous plants with workers. • Identify workers who are known to be allergic to poison ivy.
	Insects/Animals	• Inspect yourself carefully for insects or ticks after being outdoors. • Spray any wasp/hornet nests with an insect repellant from a safe distance recommended by the product's manufacturer. • Do not antagonize snakes or wild animals. • Check limbs/body for insects/insect bites. • Use protective insect repellents containing 25% DEET to prevent insect bites.
Walk site and observe conditions	Slips, trips and falls	• Maintain all equipment and tools in designated areas and out of pathways. • Look for changes in elevation and grade when walking on uneven terrain. • (If applicable) Replace manhole covers securely to prevent tripping and vehicle accidents. • If ice and slick conditions are present, use salt or sand to add traction to prevent slips. • Snow can be moved away from walkways if it poses a slip hazard. • The sites must be well maintained, such as keeping the grass cut and clearly identifying any potential trip hazards.
Monitor light level at dusk	Vision Loss	• Adequate lighting must be provided.

STANDARD OPERATING PROCEDURES (SOP)
For
MEMBRANE INTERFACE PROBE SYSTEM

COLUMBIA Technologies, LLC
1448 S. Rolling Rd, Baltimore, MD 21227, 410-536-9911 Fax: 410-536-0222

STANDARD OPERATING PROCEDURES (SOP)
For
MEMBRANE INTERFACE PROBE SYSTEM

Reviewed By:	Title	Signature	Date
Doug McInnes	Laboratory Director		

1.0 BACKGROUND

The Membrane Interface Probe System (MIPS) is used for the detection and measurement of volatile organic compounds (VOC's) vertically through the subsurface. The self-heating probe, with a gas permeable membrane and dipole conductivity sensor, is advanced into the subsurface at predetermined increments. As the probe advances, the heat generated by the probe volatilizes any VOC's in the subsurface. The VOC's then pass through the membrane, and enter into the carrier gas stream. When the carrier gas reaches the surface, it is fed directly into a Shimadzu GC 14A equipped with a PID, an ECD and an FID. The response is then recorded and displayed graphically along with the conductivity measurements.

1.1 APPLICATIONS

The standard flow rate for the nitrogen within the transfer line is 40 mL/min. This can vary depending on the ambient air temperatures and the approximate concentrations of the contamination. If the ambient air is 40 degrees or lower, the flow should be increased to 50 mL/min. If the site investigation is focused on locating lower concentrations such as defining the outer edge of a plume, or the contaminant of concern is a known "low responder", the flows can be adjusted to be within 20-30 mL/min as deemed appropriate by the operator. When looking for high levels of contamination in source areas or NAPLs, the flow may be increased to be within 50-60 mL/min.

2.0 SETUP

When looking at the back of the controller box, on the right hand side, there is a port labeled Nitrogen Source. This is where the nitrogen supply enters the system. To the left of the nitrogen source is the input for detector 1 and detector 2. The green connector, which is the detector electrometers, is inserted here.

The trunk line is what connects the MIP unit to the controller box and the GC; the following assumes that the trunk line is already connected to the MIP. The trunk line consists of 5 wires, and 2-100 foot lengths of 1/16" Teflon tubing. The brown wire is the

thermocouple wire and is made up of 2 individual wires, red and yellow. These two wires are connected to the male thermocouple connector (yellow). The red wire goes to the negative pole, and yellow to the positive: the +/- is clearly marked on the connector itself. The male thermocouple connector then gets inserted into the female connector labeled *Thermocouple* at the back of the controller box. The remaining 4 wires are associated with the *Heater and Conductivity* connector. The probe heater wires (both yellow) are connected to the top 2 lugs of this connector. The Dipole soil conductivity wires (red/white) are connected to the bottom 2 lugs of the connector. The 2 lengths of Teflon tubing should always be joined by a female-to-female adaptor when not attached to the controller box and GC. This is to prevent any particulate matter from getting inside the tubing and potentially causing a clog. The first length of tubing is always connected to the port labeled *Regulated Out* at the back of the controller box first. This is so that any particulate matter that may have entered into the tubing is expelled, so be sure that the nitrogen is flowing through the controller box. The second length of tubing is then connected to the inlet of the dryer tube. Remember that when attaching the gas lines they are to be hand tight and then a ¼ turn with a wrench.

3.0 START UP

- Fill the generator with unleaded gasoline and check engine oil
- Turn on generator and flip circuit breaker to “on”
- Plug in extension cord, and use bungi cord to secure to top of generator
- Check to ensure that nitrogen has greater than 400 psi, replace tank if lower
- Turn on nitrogen source, and check that regulator reads 60 psi out
- Open prime regulator on MIP controller box
- Turn on GC, press start on keypad, column and injector are set to ambient, while detector is set to 300 degrees
- Turn power on to MIP controller box
- Turn on power and heater for PID lamp
- Select your detectors, by changing the electrometer at the rear of the GC.
- If working with the FID, plug in air compressor and turn on hydrogen (60 psi)
- Light FID
- Take out Rod rack.
- Check flows and membrane…section 5.1
- Turn on the probe heater
- Turn on laptop and complete the parallel port connection
- Open MIP software.
- Response check…section 5.2

3.0 LOGGING PROCEDURES

- Use a rotary drill steel, or pre-probe punch to create a pilot hole if going through asphalt or concrete.
- Place the rod wiper under the foot of the probe, and line it up with the pilot hole.
- Set anchor system, if needed…section 5.3
- Put the slotted drive cap on the probe drive head, and insert the probe into the hole in the rod wiper, so that the tip of the probe is even with the ground.
- Connect the umbilical and string-pot…section 5.4
- Check the pressure readings, temperature and detector baselines.
- Press trigger to on position and commence probing.
- Push the probe 1-foot, and then wait 1 minute.
- Continue at this rate, until probe temperature can not recover to above 100 C. Extend the hold time to achieve minimum temperature if required.
- If necessary change the attenuation of the GC and the MIP if the response begins to approach the end of the current range. (see section 5.2)
- If temperature recovery improves, resume the 1-foot for 1 minute rate.
- If the contaminant is known to at or below a certain depth, it is ok to push straight to that depth without stopping. The log will still supply accurate depth and conductivity data.
- When looking for NAPL, it may be useful to push at 6-inch intervals.
- Once the target depth has been reached, wait the necessary 1 minute and turn the trigger “off”.
- Release the string-pot string from the counter weight, and disconnect the umbilical.
- Press F5 on the computer to end the log and save the data.
- Exit out of the MIP software and open the Display log.
- Recall the appropriate file, adjust the scaling to the predetermined uniform scale.
- Print out MIP log

4.0 LOGGING PROCEDURES (cont.)

- Start pulling the rods out of the ground, and replace on cart.
- When all the rods are out and on the rack, decon with Liquinox and water.
- Decon the rod wiper.
- If high levels were encountered, it may be useful to clean the membrane with methanol.
- Prior to the next location, visually inspect the probe and tighten if necessary, check mass flows and temperatures.

- If moving system to perform another log, prepare equipment for travel and repeat the above procedures. If there are no more logs to perform shutdown the system…section 5.6

5.1 FLOW TESTS

There are several critical flows that need to be monitored, and possibly adjusted to maximize the efficiency of the MIP system. These include the pressure from the supply tank to the prime regulator, the flow rate throughout the transfer line, the mass flow, and the flow rate inside the dryer tube. This is done at the beginning of everyday, and again if deemed necessary by **Columbia**'s analyst due to changing field conditions.

- The prime regulator on the front of the MIP controller box needs to be set to 20 psi using a small flathead screwdriver.
- The mass flow should be adjusted to 5 using the dial under the gauge.
- Fill the sample bulb of the flow meter half way with snoop.
- Disconnect the nitrogen line at the inlet of the dryer tube, and attach a 1/16" female-to-female adaptor.
- Insert adaptor into the flow meter line.
- Squeeze sample bulb gently to produce a single bubble.
- Adjust the mass flow dial until the meter reads 40 mL/min.
- Lock the mass flow dial in place, and record both mass flow and dial setting.
- Disconnect the nitrogen line, and attach the meter to the outlet of the dryer tube.
- The measured flow out of the Dryer tube should be 80 mL/min.
- Adjust the flow accordingly using the control valve on the opposite end of the dryer tube.
- Disconnect the meter, and turn the power off.
- The remaining volume of snoop in the sample bulb should then be poured onto the membrane to check for leaks.
- If bubbles appear around the perimeter of the membrane, use membrane wrench to tighten, and check again.
- If excessive leak at center of membrane, replace membrane…section 5.5
- Put the meter away.

5.2 RESPONSE CHECK and TRIP TIME

Although the MIP system cannot be calibrated, the system can be monitored for reproducibility and proper performance. Using detector specific compounds, a response check is performed before every MIP log. This procedure can performed using solvent vapors (a "Response Test"), or using aqueous solutions of known compounds (a "Performance Test").

RESPONSE TEST: This is done by introducing the headspace of a neat organic compound to the membrane, and then measuring the response against pre-set acceptable limits. The "trip time", or the time it takes for a mass to move across the membrane and cause a detector response, needs to be measured at the time of a response check.

- Scroll the MIP software up to view the response vs. time screen
- Hit F-1 to bring up attenuation screen, change D1 and D2 to a value of 10
- On the GC control pad, to change the range from 1 to 10 for PID/FID press DET-1-enter, Range-1-enter. For the ECD, press DET-3-enter, Range-1-enter.
- To determine the trip time, introduce Butane from a lighter into the membrane, while simultaneously starting a stopwatch.
- Record the time it takes from when the butane enters the membrane to when you first see a response on the screen.
- This value is entered into the MIP software on the main screen when starting a new log, and recorded in the logbook.
- Continue with the response checks by choosing the appropriate compound. For the PID use neat benzene, the ECD use neat trichloroethylene and for the FID use the butane from a lighter.
- Introduce the vapor for 4 seconds, then wait 7, and repeat 2 more times.
- Record the response that appears on the screen.
- Repeat the above for each detector
- The response needs to be greater than 1E+6 mV.
- When checks are complete, resent to the attenuations to the appropriate levels.

PERFORMANCE TEST: A performance test is used to evaluate detector response from target compounds in aqueous solution. Standard compounds such as Benzene and TCE are also commonly used for performance tests, but specific target compounds for a site may be used as well. The "trip time", or the time it takes for a mass to move across the membrane and cause a detector response, needs to be measured at the time of a performance check.

- Scroll the MIP software up to view the response vs. time screen
- Hit F-1 to bring up attenuation screen, change D1 and D2 to a value of 10
- On the GC control pad, to change the range from 1 to 10 for PID/FID press DET-1-enter, Range-1-enter. For the ECD, press DET-3-enter, Range-1-enter.
- To determine the trip time, introduce Butane from a lighter into the membrane, while simultaneously starting a stopwatch.
- Record the time it takes from when the butane enters the membrane to when you first see a response on the screen.
- This value is entered into the MIP software on the main screen when starting a new log, and recorded in the logbook.
- Continue with the performance checks by choosing the appropriate compound. For the PID use a benzene solution (or other site-specific target compound), the

ECD use a trichloroethylene solution (or other site-specific compounds). The FID response is still evaluated using butane from a lighter unless some site-specific compound is chosen for testing.

- Prepare Stock Standard of compound(s) of interest (see MIPS Performance Test Solution Prep spreadsheet)
- Immerse the probe in a container of clean water (commonly a 2" PVC tube) to stabilize the baseline.
- Check the stability of the detector vs time data on the MIP software
- Prepare 500 ml Testing standard from Stock Standards, place in 2" PVC Tube.
- Insert the probe into the test solution of known concentration for 45 seconds
- Return the probe to the tube containing clean water
- Record trip time and response for each detector in field notes
- Compare results to previous measurements
- If the result varies more than 50% for any detector, begin trouble-shooting evaluation. Note any corrective actions performed in field notes
- When checks are complete, resent to the attenuations to the appropriate levels.

5.3 ANCHOR SYSTEM

Due to the sensitivity of the probe membrane, an anchoring system is recommended to push the MIP and rods into the subsurface, so to avoid using the hammer and potentially damaging the MIP down-hole. Using a centered starting position, move the Geoprobe unit to the extreme left, before bringing it 4 inches back towards center. This is the location that the first anchor will be set. Using the rotation feature of the Geoprobe 5400, turn the anchor to depth, leaving just the stem visible above the ground surface. To set the other two anchors, swing the Geoprobe unit to the extreme right position before bringing it 4 inches back towards center, then swing it back to centered starting position, extend the Geoprobe unit to the extreme forward position and then back 4 inches. Place the anchor plate over the foot of the Geoprobe, and the 3 auger stems. It is very important that when setting the plate that the probe's vertical movement of the hammer is not obstructed. The chain vices are then secured to the auger flights, which will lock the anchor plate to the foot of the probe.

5.4 STRINGPOT

Attach the string-pot to the string-pot bracket, and then to the main anchoring bolt of the probe hammer. The string-pot bottom clamp must then be bolted to the foot of the hammer. Prior to operation, the cotter pin is to be removed from the foot bracket so that the counter weight is free to move, and then inserted into the eyebolt on top of the

counter weight. When the string or cable connects these two devices, the cable should be parallel to the probe and perpendicular to the ground. It is essential that the string-pot cable be connected to the counter weight prior to the activation of the trigger, or the depth measurements will not be accurate. The umbilical should be attached to the string-pot before the activation of the trigger as well.

5.5 MEMBRANE REPLACEMENT

It is very important to note that while completing the following procedure to use great care when screwing in the new membrane. Be sure that the threads do not become cross-threaded. This would make a complete seal impossible, which will then greatly hinder the performance of the MIP system.

- Secure the probe in a vice.
- Clean the membrane and surrounding area thoroughly.
- Using the dental pick, clean out the 4 holes in the membrane.
- Using the membrane wrench, carefully unscrew the membrane while applying equal pressure to the top of the membrane.
- Remove the membrane, and use the pick to clean up the interior threads of the probe, while blowing out the freed dirt.
- Remove the washer carefully so to not allow any dirt to fall into the chamber.
- Insert a new washer
- Thread the new membrane into position, and tighten with the membrane wrench.
- Using Snoop, check for leaks around the perimeter of the membrane.
- If bubbles appear around the edge, use the wrench to tighten more.
- Continue to tighten, until there is a complete seal.
- There will be a certain amount of gas escaping through the center of the membrane; this is acceptable. It usually takes about 75 to 100 feet to fully condition the membrane.

5.6 SHUTDOWN PROCEDURES

- Turn off the power supply to the heater.
- Turn off the PID lamp and heater.
- Turn off GC.
- If using FID, close the valve to the tank of hydrogen.
- When the probe temperature has returned to ambient, turn off power to MIP controller box.
- Close the prime regulator on MIP controller box.
- Close the valve to the tank of nitrogen.
- Shutdown the computer.
- Shutdown the generator, and close valve to gas line
- Put everything back where you found it.

LOAD LIST

Quantity	Item Description	Part Number	Vendor
2	Membrane Interface Probe	MP3510	Geoprobe
5	Replacement Membrane	MP3512	Geoprobe
2	Trunk line (Transfer line)	MP2550	Geoprobe
2	String-pot	SC160	Geoprobe
1	String-pot Mounting Bracket	SC110	Geoprobe
1	String-pot Mounting Bracket for 66DT	11751	Geoprobe
1	String-pot Bottom Clamp	SC111	Geoprobe
1	String-pot Piston Weight	SC112	Geoprobe
2	String-pot Cordset (umbilical)	SC161	Geoprobe
5	Gortex Strip	12138	Geoprobe
2	Slotted Drive Cap	AT1202	Geoprobe
2	Slotted Pull Cap	AT1203	Geoprobe
1	Rod Wiper	AT1255	Geoprobe
2	LB Sample Tube (wire cavity)	AT6621	Geoprobe
2	Adaptor, 1.25”->1.375”	MP2512	Geoprobe
2	SP Drive Head, 1.375	GW1516	Geoprobe
1	Membrane Wrench	16172	Geoprobe
1	O-ring Pick	AT102	Geoprobe
1	MIP Kit (O-rings, 1/16” ferrules & nuts, wirenuts, Silcosteel)		Geoprobe
3	4 foot x 4 inch auger	10245	Geoprobe
3	Chain Vice	10075	Geoprobe
3	Auger Plates	10176	Geoprobe
1	Anchor Plate	15340	Geoprobe
1	Tool Box		
1	100 Ft. Extension Cord		
1	Space Heater		
1	Road Atlas		
1	Generator		
1	Spare Gas Can		
1	Quart of Motor Oil		
1	Volt Meter		
1	Snoop		
1	Flow Meter		
1	9-Volt Battery		

LOAD LIST (cont)

Quantity	Item Description	Part Number	Vendor
5	1.44 Floppy Disk		
1	Printer		
1	Ream of paper		
1	Spare Ink Cartridge		
1	Power/Parallel Cords		
1	Printer Drivers on Disk		
1	Cylinder of nitrogen		
1	Cylinder of hydrogen		
2	Regulators		
2	Box of Nitrile Gloves		
1	Lap Top w/ Software		
1	Air Pump		
1	Air Compressor		
1	Fire Extinguisher		
1	Spare PID Lamp		
1	Neat Standards…mecl2, Toluene		
1	Site Specific Neat Standards		
1	Butane Lighter		
1	Hard Hat		
1	Pair of Safety Glasses		
1	Ear Protection		
1	Steel Toe Boots		
1	OSHA 8 hr/ Medical Clearance		

MIPS PERFORMANCE TEST SOLUTION PREP

50 MG/ML STOCK -- 100, 10, AND 1 MG/L (PPM) SOLUTIONS IN 500 ML DI

Compound Name	Density	25 ml of 50 mg/mL Stock from neat in 25 ml MEOH	10 ml of 50 mg/mL Stock from neat in 10 ml MEOH	Volume 0f 50 mg/L Stock for 100 mg/L in 500 ml DI	Volume 0f 50 mg/L Stock for 10 mg/L in 500 ml DI	Volume 0f 50 mg/L Stock for 1 mg/L in 500 ml DI
	(g/L)	(uL)	(uL)	(uL)	(uL)	(uL)
Trichloroethylene	1.4642	854	341	1000	100	10
Methylene Chloride	1.33	940	376	1000	100	10
1,2 Dichloroethylene	1.27	984	394	1000	100	10
1,1-Dichloroethylene	1.213	1031	412	1000	100	10
1,1,2,2-Tetrachloroethane	1.586	788	315	1000	100	10
Benzene	0.8765	1426	570	1000	100	10
Toluene	0.87	1437	575	1000	100	10
Tetrachloroethylene	1.6227	770	308	1000	100	10
Carbon Tetrachloride	1.594	784	314	1000	100	10
Chlorobenzene	1.106	1130	452	1000	100	10
1,1,1-Trichloroethane	1.3376	935	374	1000	100	10
1,1,2-Trichloroethane	1.442	867	347	1000	100	10
1,1-Dichloroethene	1.2129	1031	412	1000	100	10
1,4-Dichlorobenzene	1.241	1007	403	1000	100	10
MTBE	0.7404	1688	675	1000	100	10
Hexanes	0.6603	1893	757	1000	100	10
MEK (2-butanone)	0.81	1543	617	1000	100	10
Ethylbenzene	0.87	1437	575	1000	100	10
m-Xylene	0.86	1453	581	1000	100	10
o-Xylene	0.88	1420	568	1000	100	10
p-Xylene	0.86	1453	581	1000	100	10
Methylene Chloride	1.33	940	376	1000	100	10
Diesel	0.81	1543	617	1000	100	10
Naphthalene	1.15	1087	435	1000	100	10
Acetone	0.79	1582	633	1000	100	10
1,2-Dichloroethane	1.24	1008	403	1000	100	10
Trichlorfluoromethane	1.374	910	364	1000	100	10
1,1-Dichloroethane	1.18	1059	424	1000	100	10
1,2-Dibromoethane	2.1	595	238	1000	100	10
tert_Butyl Alcohol (TBA)	0.79	1582	633	1000	100	10

Tetra Tech NUS, Inc.

BORING LOG

PROJECT NAME: _____ BORING No.: _____
PROJECT NUMBER: _____ DATE: _____
DRILLING COMPANY: _____ GEOLOGIST: _____
DRILLING RIG: _____ DRILLER: _____

Sample No. and Type or RQD	Depth (Ft.) or Run No.	Blows / 6" or RQD (%)	Sample Recovery / Sample Length	Lithology Change (Depth/Ft.) or Screened Interval	MATERIAL DESCRIPTION: Soil Density/ Consistency or Rock Hardness	MATERIAL DESCRIPTION: Color	MATERIAL DESCRIPTION: Material Classification	USCS *	Remarks	PID/FID Reading (ppm): Sample	PID/FID Reading (ppm): Sampler BZ	PID/FID Reading (ppm): Borehole**	PID/FID Reading (ppm): Driller BZ**

* When rock coring, enter rock brokeness.

** Include monitor reading in 6 foot intervals @ borehole. Increase reading frequency if elevated reponse read.

Drilling Area Background (ppm): _____

Remarks: _____

Converted to Well: Yes _____ No _____ Well I.D. #: _____

Tetra Tech NUS, Inc.

PROJECT: _____ **JOB #:** _____
LOCATION: _____ **DATE:** _____
PROJECT MANAGER: _____ **FOL:** _____

DAILY ACTIVITIES CHECKLIST

Startup Checklist

Activity	Yes	No	N/A
Pertinent site activities/information entered into site logbook			
All onsite personnel listed in logbook			
Required medical information onsite for all workers (TtNUS and Subcontractors)			
Required MSDS's onsite			
Proper equipment calibrations performed (list equipment)			
1 _____			
2 _____			
3 _____			
4 _____			
Calibration logs filled out			
Tailgate H&S meeting held prior to beginning field activities			
Required work permits filled out/signed			
Required utility clearances obtained			
Required PPE onsite and in use			
Information required to be posted is in place (OSHA poster, hospital route, key phone numbers, etc.)			

Exit Checklist

Activity	Yes	No	N/A
Logbooks completely and comprehensively filled out			
Field forms complete and accounted for/properly filed			
Samples properly packaged/shipped			
COCs faxed to appropriate in-house personnel			
All equipment accounted for, on charge if needed, and properly secured			
All personnel accounted for			
Arrangements made for upcoming work (permits, clearances, equipment, etc.)			
Site properly secured			

Note - not all items listed apply to every job, and some additional requirements may apply on a job-specific basis.

Tetra Tech NUS, Inc.

EQUIPMENT CALIBRATION LOG

PROJECT NAME : ____________________ INSTRUMENT NAME/MODEL: ____________________

SITE NAME: ____________________ MANUFACTURER: ____________________

PROJECT No.: ____________________ SERIAL NUMBER: ____________________

Date of Calibration	Instrument I.D. Number	Person Performing Calibration	Instrument Settings		Instrument Readings		Calibration Standard (Lot No.)	Remarks and Comments
			Pre-calibration	Post-calibration	Pre-calibration	Post-calibration		

Tetra Tech NUS, Inc.

GROUNDWATER LEVEL MEASUREMENT SHEET

Project Name: _____ **Project No.:** _____

Location: _____ **Personnel:** _____

Weather Conditions: _____ **Measuring Device:** _____

Tidally Influenced: **Yes** ____ **No** ____ **Remarks:** _____

Well or Piezometer Number	Date	Time	Elevation of Reference Point (feet)*	Total Well Depth (feet)*	Water Level Indicator Reading (feet)*	Thickness of Free Product (feet)*	Groundwater Elevation (feet)*	Comments

* All measurements to the nearest 0.01 foot

Tetra Tech NUS, Inc.

GROUNDWATER SAMPLE LOG SHEET

Project Site Name: _____
Project No.: _____

[] Domestic Well Data
[] Monitoring Well Data
[] Other Well Type: _____
[] QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.O.C. No.: _____
Type of Sample:
[] Low Concentration
[] High Concentration

SAMPLING DATA:

Date: / Time:	**Color** (Visual)	**pH** (S.U.)	**S.C.** (mS/cm)	**Temp.** (^{0}C)	**Turbidity** (NTU)	**DO** (mg/l)	**Salinity** (%)	Other
Method:								

PURGE DATA:

Date:	**Volume**	**pH**	**S.C.**	**Temp.**	**Turbidity**	**DO**	**Salinity**	Other
Method:								
Monitor Reading (ppm):								
Well Casing Diameter & Material Type:								
Total Well Depth (TD):								
Static Water Level (WL):								
One Casing Volume(gal/L):								
Start Purge (hrs):								
End Purge (hrs):								
Total Purge Time (min):								
Total Vol. Purged (gal/L):								

SAMPLE COLLECTION INFORMATION:

Analysis	**Preservative**	**Container Requirements**	**Collected**

OBSERVATIONS / NOTES:

Circle if Applicable:

MS/MSD | **Duplicate ID No.:**

Signature(s):

CHAIN-OF-CUSTODY RECORD

Mobile Laboratory Services

☐ 25132 SW 1st Avenue
Newberry, FL 32669
TEL (352) 367-0073
FAX (352) 472-5832

☐ 200 Quade Drive
Cary, NC 27513
TEL (919) 678-0030

MOBILE UNIT #

CLIENT NAME	PROJECT NAME & ADDRESS														
SAMPLERS	CONTACT PERSON						BATCH # **(Lab Use Only)**								
SAMPLE FIELD ID.\ NUMBER	DATE SAMPLED	TIME SAMPLED	COMP.	GRAB	DATE REC'D	TIME REC'D	STATION LOCATION / No.	SAMPLE MATRIX	NUMBER OF CONTAINERS	IDENTIFY PARAMETERS DESIRED AND NO. OF CONTAINERS / VOLATILES					**COMMENT / SAMPLE PRE FIX**

PRESERVATION
C Chilled
H HCL
Ot Other (see Remarks)

Precleaned Containers Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*	Date / Time	**Remarks and Observations**
Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*	Date / Time	

Matrix Types S Soil SW Surface Water GW Ground Water SG Soil Gas

LOW FLOW PURGE DATA SHEET

PROJECT SITE NAME: _____ WELL ID.: _____

PROJECT NUMBER: _____ DATE: _____

Time (Hrs.)	Water Level (Ft. below TOC)	Flow (mL/Min.)	pH (S.U.)	S. Cond. (mS/cm)	Turb. (NTU)	DO (mg/L)	Temp. (Celcius)	ORP mV	Salinity % or ppt	Comments

SIGNATURE(S): _____ PAGE___OF___

Tetra Tech NUS, Inc.

MONITORING WELL DEVELOPMENT RECORD

Site: _____ Depth to Bottom (ft.): _____ Project Name: _____
Well: _____ Static Water Level Before (ft.): _____ Project Number: _____
Date Installed: _____ Static Water Level After (ft.): _____ Site Geologist: _____
Date Developed: _____ Screen Length (ft.): _____ Drilling Co.: _____
Dev. Method: _____ Specific Capacity: _____
Pump Type: _____ Casing ID (in.): _____

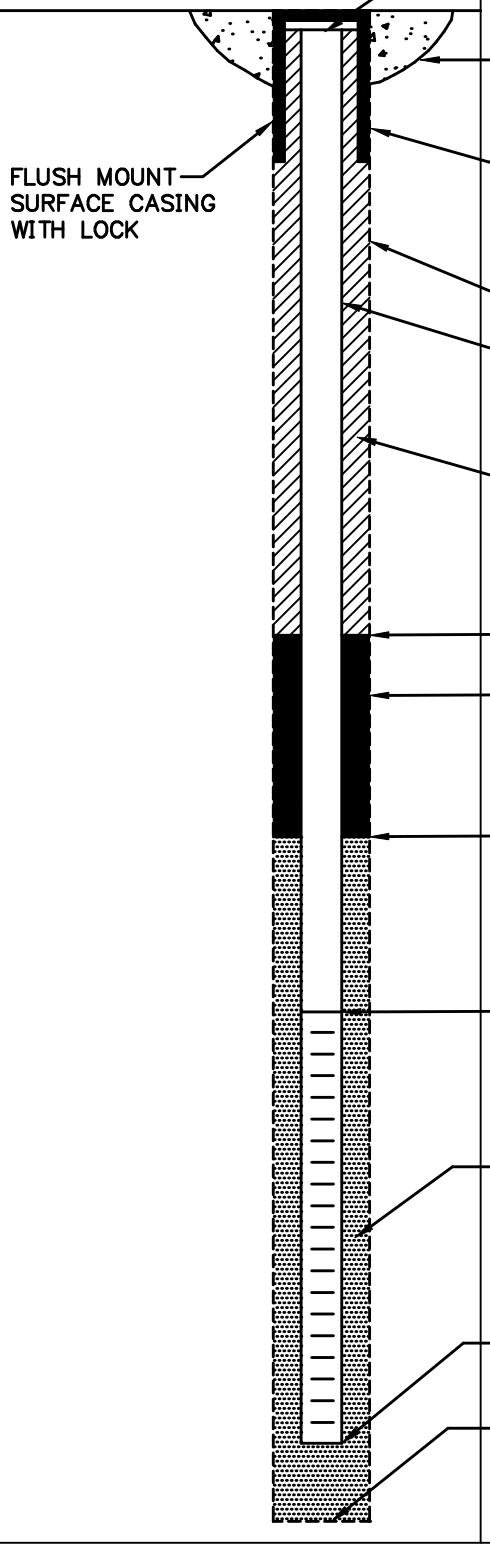
Time	Estimated Sediment Thickness (Ft.)	Cumulative Water Volume (Gal.)	Water Level Readings (Ft. below TOC)	Temperature (Degrees C)	pH	Specific Conductance (Units _____)	Turbidity (NTU)	Remarks (odor, color, etc.)

WELL NO.: _____

Tetra Tech NUS, Inc.

OVERBURDEN
MONITORING WELL SHEET
FLUSH - MOUNT

PROJECT _____	LOCATION _____	DRILLER _____
PROJECT NO. _____	BORING _____	DRILLING METHOD _____
DATE BEGUN _____	DATE COMPLETED _____	
FIELD GEOLOGIST _____		DEVELOPMENT METHOD _____
GROUND ELEVATION _____	DATUM _____	



ELEVATION TOP OF RISER: _____

TYPE OF SURFACE SEAL: _____

_____

TYPE OF PROTECTIVE CASING: _____

_____

I.D. OF PROTECTIVE CASING: _____

DIAMETER OF HOLE: _____

TYPE OF RISER PIPE: _____

_____

RISER PIPE I.D.: _____

TYPE OF BACKFILL/SEAL: _____

_____

ELEVATION/DEPTH TOP OF SEAL: _____ / _____

TYPE OF SEAL: _____

_____

ELEVATION/DEPTH TOP OF SAND: _____ / _____

ELEVATION/DEPTH TOP OF SCREEN: _____ / _____

TYPE OF SCREEN: _____

SLOT SIZE x LENGTH: _____

TYPE OF SAND PACK: _____

_____

DIAMETER OF HOLE IN BEDROCK: _____

ELEVATION / DEPTH BOTTOM OF SCREEN: _____ / _____

ELEVATION / DEPTH BOTTOM OF SAND: _____ / _____

ELEVATION/DEPTH BOTTOM OF HOLE: _____ / _____

BACKFILL MATERIAL BELOW SAND: _____

_____

ACAD: FORM_MWFM.dwg 07/20/99 INL

Tetra Tech NUS, Inc.

PROJECT: _____ **LOCATION:** _____

JOB & CTO #: _____ **MOBILIZATION DATE:** _____

PROJECT MANAGER: _____ **RETURN DATE:** _____

FIELD PROJECT PRE-MOBILIZATION CHECKLIST

TRAVEL

___ Airline reservations
___ Hotel reservations/BOQs
___ Vehicle rental
___ Itinerary
___ Phone/pager number

DRILLING/DPT/SURVEY

Subcontractor

___ POC phone #/address
___ Drill Specification RFP
___ Contact (time & place to meet)
___ Confirm subcontract w/ TtNUS Procurement
___ Health and Safety documentation for all personnel on site
___ Copy of Drillers license
___ Well / boring permits

Utilities (2 weeks lead time)

___ Contact Site POC (Date:_____)
___ Contact Local "Call Before You Dig"
___ Utility Clearance Form

Forms

___ Boring logs / Test Pit logs
___ Well construction / development forms
___ Daily activity forms
___ IDW inventory
___ IDW drum labels
___ Chemical Inventory
___ MSDS's

EQUIPMENT MOBILIZATION

___ Equipment Requisition form completed / equipment ordered
___ 3rd Party rental / misc. equipment ordered
___ Equipment calibration forms
___ Span / calibration gas and regulator

SAMPLING

Forms

___ Sample log sheets
___ Low-flow purge data sheets
___ COC records
___ COC seals
___ Sample labels (from database group)

Laboratory

___ POC address/phone#
___ Order bottles / preservatives
___ Shipping address, also check Sat. address
___ Bottle & preservation req'ts from lab
___ _____

MISCELLANEOUS

Schedule

___ Plan field operations w/ Project manager

Documents for Field Program

___ Logbook(s)
___ Field Sampling plan
___ Health & Safety plan
___ Maps
___ H & S Guidance Manual

Authorization

___ Kick-off meeting held
___ Gov't rate letter
___ H&S/OSHA 40-hour certifcate
___ 8-Hour Refresher Training Certificate
___ Medical Clearance Letter
___ Supervisory Training Certificate
___ Health & Safety Clearance Letter
___ Full-size OSHA Poster

HYDROGEOLOGY EQUIPMENT

___ Slug test/pumping test forms
___ Groundwater elevation data sheets
___ Graph paper
___ Data Logger/transducer/data cable
___ Existing well construction & water level data
___ M-Scope, slug

SHIPPING

Forms

___ FedEx Airbills, local dropoff location & hours
___ FedEx Gov. Acct# (1771-8058-0)
___ Lab Shipping Labels
___ Warehouse Shipping Labels
___ Blank Labels

Supplies

___ Tape
___ Packing materials
___ Baggies, Large garbage bags

OTHER

___ Site POC name/phone #
___ Personnel information to POC
___ Mobilization schedule to POC
___ Site access authorizations
___ Field office / trailer arrangements made
___ Electric, phone hookups arranged
___ Steel-toed boots, safety glasses, & hard hat
___ First aid equipment
___ Insect repellent
___ _____
___ _____

Note - not all items listed apply to every job, and some additional requirements may apply on a job-specific basis.

Tetra Tech NUS, Inc.

QA SAMPLE LOG SHEET

Project Site Name: _____ Sample ID Number: _____
Project Number: _____ Sampled By: _____
Sample Location: _____ C.O.C. Number: _____
QA Sample Type:

[] Trip Blank [] Rinsate Blank
[] Source Water Blank [] Other Blank _____

SAMPLING DATA:

Date: _____
Time: _____
Method: _____

WATER SOURCE:

[] Laboratory Prepared [] Tap
[] Purchased [] Fire Hydrant
[] Other _____

PURCHASED WATER INFORMATION (If Applicable as Source or Rinsate Water):

Product Name: _____
Supplier: _____
Manufacturer: _____
Order Number: _____
Lot Number: _____
Expiration Date: _____

RINSATE INFORMATION (If Applicable):

Media Type: _____
Equipment Used: _____
Equipment Type:
[] Dedicated
[] Reusable

SAMPLE COLLECTION INFORMATION:

Analysis	Preservative	Container Requirements	Collected
Volatiles	Cool 4°C & HCl		YES / NO
Semivolatiles	Cool 4°C		YES / NO
Pesticide / PCB	Cool 4°C		YES / NO
Metals	Cool 4°C & HNO_3		YES / NO
Cyanide	Cool 4°C & NaOH		YES / NO

OBSERVATIONS / NOTES:

Signature(s):

Tetra Tech NUS, Inc.

SOIL & SEDIMENT SAMPLE LOG SHEET

Project Site Name: _____
Project No.: _____

[] Surface Soil
[] Subsurface Soil
[] Sediment
[] Other: _____
[] QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.O.C. No.: _____

Type of Sample:
[] Low Concentration
[] High Concentration

GRAB SAMPLE DATA:

Date: / Time: / Method: / Monitor Reading (ppm):	Depth Interval	Color	Description (Sand, Silt, Clay, Moisture, etc.)

COMPOSITE SAMPLE DATA:

Date: / Method: / Monitor Readings (Range in ppm):	Time	Depth Interval	Color	Description (Sand, Silt, Clay, Moisture, etc.)

SAMPLE COLLECTION INFORMATION:

Analysis	Container Requirements	Collected	Other

OBSERVATIONS / NOTES:

MAP:

Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

APPENDIX B

ANALYTICAL LABORATORY SOPS AND ACCREDITATIONS

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-202
Revision History
Cover Page
Page 1

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

Prepared By: GC/MS Group Date: 2/97

Approved By:

Group Supervisor: JA Halay Date: 011201

Operations Manager: for C. Burton Date: 1/15/01

QA Officer: Deborah J. Nadeau Date: 1·23·01

General Manager: Deborah F. Nadeau Date: 1/16/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
03 8260B	Format changes, added pollution prevention, changes to calibration section, new limits, added instrument. Other minor changes throughout.	DN	1·23·01	1·23·01
04 8260B	Revised sections 7.5.3.1, 7.5.5, 7.7.1, 7.8.2 + Table 2 to comply with South Carolina. Added NH oxygenates to calibration.	DN	5·23·01	5·23·01
05 8260B	Updated VOA calibration standard mixes. Added statistical limits for LCS/MS/MSD recoveries and the updated corrective actions	DN	5·21·02	5·21·02
06 8260B	Reorganization of sections 4, 5, 6 and 7, and Tables and Figures. Added definitions and information for the new data processing system.	MRC	05.03.04	05.03.04
07 8260B	minor changes rewording of sec 1.7.6.3 preservation of calcareous soils	LAD	020305	020305

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
08 8260B	Added references, setup and operation for the Encon / Centurion autosampler / Purge and Trap. Added ref. to instrument "T" and removed instrument "Q". Edited Std. conc. to reflect new instrumentation. Minor changes throughout to reflect current practice and correct typos.	LAD	04/06	04/06
09 8260B	Sect. 4H - added list of waste streams generated and location of satellites. Clarified RT window studies. Added reference to MI Sop. Removed Grand mean Calibration model. Added wording for project specific acceptance criteria. Added LCS marginal outlier criteria. Added wording clarifying calibration verification Std. Criteria and corrective action. Reworded Correlation	LAD	LAD 7-25-07 ~~03/07~~ 07/07	~~03/07~~ 07/07
10	Coefficient criteria Updated Sections 7.4.5, 7.4.6, 7.4.7, 7.5.2, 8.1, 10.0 and Table 1 with DoD QSM version 4.1 criteria	LAD	08/09	08/09
11	Added Table 2 with DoD QSM V. 4.1 QC Requirements. Added if the MSD Batch requirement cannot be fulfilled, a LCSD must be analyzed. Removed "2" instrument and added the "C" and "D" instruments.	LAD	04/10	04/10

TITLE: **ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-202-11**, titled **ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-202-11**, titled **ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**.

Recipient: _____ Date: _____

TITLE: **ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures utilized by Katahdin Analytical Services, Inc. laboratory personnel to prepare and analyze aqueous and solid matrix samples for purgeable organics by GC/MS in accordance with SW-846 Method 8260, current revision.

This SOP will consolidate all aspects of the analyses in one working document, to be revised as necessary, for the purposes of consistency in data quality.

1.1 Definitions

VOC: Volatile Organic Compounds

VOA: Volatile Organic Analysis

ANALYTICAL BATCH: 20 or fewer samples that are analyzed together with the same method sequence and the same lots of reagents and with the handling practices common to each sample within the same time period or in continuous sequential time periods.

METHOD BLANK (laboratory reagent blank): A quality control sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. Laboratory reagent grade water is used as a blank matrix. The blank is taken through the appropriate steps of the process.

CALIBRATION CHECK: Verification of the ratio of instrument response to analyte amount, a calibration check is done by analyzing a mid point standard. The calibration check verifies that instrument conditions are sufficiently similar to those at initial calibration.

CALIBRATION STANDARD (WORKING STANDARD): A solution prepared from the stock standard solution that is used to calibrate the instrument response with respect to analyte concentration.

INDEPENDANT CALIBRATION STANDARD: A solution prepared from a stock standard solution independent of the standard that is used to calibrate the instrument. This is prepared as an LCS and analyzed after the calibration before any sample analysis.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control and to measure the degree of accuracy of the determination.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions containing target analytes are added to a sample matrix prior to sample

extraction, in the case of soils, and/or analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the spiked analytes. The relative percent difference between the samples is calculated and used to assess analytical precision.

STANDARD CURVE (CALIBRATION CURVE): A curve that plots concentration of known analyte standard versus the instrument response to the analyte.

STOCK STANDARD SOLUTION: A concentrated solution containing a single analyte or mix of certified standards, or a concentrated solution of a single analyte prepared in the laboratory with an assay reference compound. Stock standard solutions are used to prepare calibration standards.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition as well as extraction and chromatography characteristics, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate. Surrogates provide an indication of the accuracy for the analytical determination in a discrete sample matrix.

TARGET: A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

TARGET DB: An oracle database used to store and organize all Target data files.

QUICKFORMS: A laboratory reporting software for Target and Target DB. The QuickForms report module for Target is preconfigured with generalized forms and US EPA CLP report forms and disk deliverables, which can be customized.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of volatile organics by the current revision of EPA Method 8260. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training and Demonstration of Capability”.

It is the responsibility of all Katahdin technical personnel involved in analysis of volatile organics by Method 8260 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate logbook. Any deviations from the test or irregularities with the samples should also be recorded in the lab logbook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

After analysis, partially-filled VOA vials and sample jars are returned to the appropriate refrigerators to be disposed of in adherence with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, Sample Disposal, current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP SD-903.

Sample aliquots used for analysis are disposed of in accordance with SOP SD-903 and the Katahdin Hazardous Waste Management Plan and Safety Manual. The soil samples must be decanted and the soil fraction disposed of separately in compliance with Katahdin's disposal policies.
There are three general types of waste generated while performing the 8260 method. The "K" waste is a combination of water, sample aliquot (post analysis), as well as internal and surrogate standards. "K" waste is generated when preparing QC, during sample analysis, and procedural cleanup. There are "K" satellites

attached to each GC/MS instrument as well as an additional satellite located adjacent to the VOA sample preparation bench. "O" waste consists of methanol (as well as trace amounts of volatile analytes) and is generated when standard preparation syringes are rinsed three times with methanol. The "O" waste stream satellite is located inside the fume hood. Organic soil waste stream "I" consists of any solid left over from sample preparation and/or analysis and is located inside the fume hood. All satellites listed above are stored in a secondary container and are located in the Volatile Organics Laboratory room 111.

2.0 SUMMARY OF METHOD

The general methodology involves purging aqueous and soil samples with helium, an inert gas, for a set period of time to efficiently transfer purgeable organics to the gaseous phase. Soil samples with higher contaminant levels are extracted with methanol prior to the helium purge. These volatile organics are then retained on a cooled trap (commercially available trap suitable for the methodology) before heating causes desorption into a gas chromatograph for compound separation. Detection occurs with an electron impact ionization mass spectrometer.

3.0 INTERFERENCES

Interfering contamination may occur when a sample containing low concentrations of VOCs is analyzed immediately after a sample containing high concentrations of VOCs. During initial data review, all analyses are evaluated for potential carryover. Any samples that have suspected carryover are reanalyzed. GC/MS policy is to reanalyze a sample with positive detects greater than the Practical Quantitation Limit (PQL) that has been run immediately after a sample with the same positive detects over the upper limit of the calibration. Typically 2 or 3 rinsing blanks are analyzed at the end of a sequence. Samples are not analyzed on the instrument until a blank with no detects above PQL can be obtained. If the lines are determined to be contaminated, then the entire Tekmar or Archon must be backflushed with warm methanol and water.

4.0 APPARATUS AND MATERIALS

4.1 GC: Hewlett Packard 6890 & 5890

4.2 Mass Spectrometers (MS): HP5973, HP5972 and HP5970

4.3 Helium: Carrier gas for routine applications. All carrier gas lines must be constructed from stainless steel or copper tubing; non-polytetrafluoroethylene (non-PTFE) thread sealant or flow controllers with rubber component are not to be used.

4.4 Columns: RTX-VMS, 40 meter, 0.18 mm ID or equivalent.

4.5 Purge and Traps: Archon 5100, Tekmar 2016 and Centurion auto samplers, and Tekmar 2000, 3000 and Encon concentrators.

4.6 Purge tubes: 5 mL fritted and 25 mL fritted purge vessels and 40 mL VOA vials for soil analysis.

4.7 Hamilton Gastight syringes: 2.00 uL to 25.00 mL.

4.8 Acquisition System: The acquisition system must be interfaced to the MS and allow continuous acquisition of data throughout the duration of the chromatographic program. It must permit, at a minimum, the output of time vs. intensity (peak height or peak area). Hewlett Packard Chemstation or equivalent.

4.9 Data System: The Target software is used for processing data and generating forms.

5.0 REAGENTS

5.1 Purge and trap grade methanol

5.2 Organic-free Laboratory reagent grade water: Siemens, Poland Spring, or equivalent. This water may need to be purged with nitrogen to eliminate organic contaminants such as Methylene chloride and Chloroform, which are commonly found at ambient levels in the laboratory.

5.3 Standards: Stock standards and working standards are received and recorded in accordance with SOP CA-106 “Standard Preparation and Documentation”.

5.3.1 The expiration date for all standards is six months from date of opening the ampule with the following exceptions:

Volatile gases expire within 2 weeks of opening ampule (gases are dichlorodifluoromethane, chloromethane, bromomethane, vinyl chloride, chloroethane, and trichlorofluoromethane).

New standards must be opened if degradation is observed.

5.3.2 Secondary dilution standards

5.3.2.1 Calibration Mix – Prepare a standard in purge and trap methanol containing the compounds listed below. The final concentration of each compound is 200 ug/mL (some individual analyte concentrations may vary, i.e. Ketones). The standard should be prepared in a 1.0 mL conical vial with a mini-inert valve cap. The standard must be prepared every 7 days and stored in the VOA standards freezer between uses.

Acetone	Dibromochloromethane	P-Isopropyltoluene
Benzene	1,2-Dibromoethane	Methylene Chloride
Bromobenzene	Dibromomethane	4-Methyl-2-Pentanone
Bromochloromethane	1,2-Dichlorobenzene	Naphthalene
Bromodichloromethane	1,3-Dichlorobenzene	N-Propylbenzene
Bromoform	1,4-Dichlorobenzene	Styrene
Bromomethane	Dichlorodifluoromethane	1,1,1,2-Tetrachloroethane
2-Butanone	1,1-Dichloroethane	1,1,2,2-Tetrachloroethane
n-Butylbenzene	1,2-Dichloroethane	Tetrachloroethene
sec-Butylbenzene	1,1-Dichloroethene	Tetrahydrofuran
tert-Butylbenzene	cis-1,2-Dichloroethene	Toluene
Carbon Disulfide	Trans-1,2-Dichloroethene	1,2,3-Trichlorobenzene
Carbon Tetrachloride	1,2-Dichloropropane	1,2,4-Trichlorobenzene
Chlorobenzene	1,3-Dichloropropane	1,1,1-Trichloroethane
Chloroethane	2,2-Dichloropropane	1,1,2-Trichloroethane
2-Chloroethylvinyl Ether	1,1-Dichloropropene	Trichloroethene
Chloroform	Cis-1,3-Dichloropropene	Trichlorofluoromethane
Chloromethane	Trans-1,3-Dichloropropene	1,2,3-Trichloropropane
2-Chlorotoluene	Ethylbezene	1,2,4-Trimethylbenzene
4-Chlorotoluene	Hexachlorobutadiene	Vinyl Acetate
Cyclohexane	2-Hexanone	Vinyl Chloride
1,2-Dibromo-3-Chloropropane	Idomethane	1,3,5-Trimethylbenzene
Isopropylbenzene	Methyl Tert-Butyl Ether	1-Chlorohexane

5.3.2.2 Extras mix – Prepare a standard as above containing the compounds listed below. The final concentration of each compound is 200 ug/mL. The standard should be prepared in a 1.0 mL conical vial with a mini-inert valve cap. The standard must be prepared every 30 days and stored in the VOA standards freezer between uses.

Acetonitrile	Isobutyl Alcohol
Acrolein	Methacrylonitrile
Acrylonitrile	Methylcyclohexane
Allyl Chloride	Methyl Acetate
Chloroprene	Methyl Methacrylate
Diethyl Ether	Methyl Tert-Butyl Ether
Cis-1,4-Dichloro-2-Butene	Pentachloroethane
Trans-1,4-Dichloro-2-Butene	Propionitrile
1,4-Dioxane	Tertiary-Amyl Methyl Ether
Di-Isopropyl Ether	Tertiary-Butyl Alcohol
Ethyl Methacrylate	1,3,5-Trichlorobenzene
Ethyl Tertiary-Butyl Ether	1,2,3-Trimethylbenzene
Freon-113	

5.3.2.3 Independent Calibration Verification Standard, Laboratory Control Spike and MS/MSD Mixture - Prepare a standard as above containing the compounds listed in Table 3. The final concentration of each compound is 200 ug/mL (some individual analyte concentrations may vary, i.e. Ketones). The standard should be prepared in a 1.0 mL conical vial with a mini-inert valve cap. The standard must be prepared every 7 days and stored in the VOA standards freezer between uses.

5.3.2.4 Surrogate Spiking Solution - Prepare a standard as above containing the compounds listed below. The final concentration of each compound is 250 ug/mL or 50 ug/mL depending on which autosampler you will be using. The standard must be prepared every 14 days and stored on the Archon and/or the Centurion autosampler in a pressurized vial or in the VOA standards freezer between uses.

4-Bromofluorobenzene
1,2-Dichloroethane-D_4
Toluene-D_8
Dibromofluoromethane

5.3.2.5 Internal Standard Solution - Prepare a standard as above containing the compounds listed below. The final concentration of each compound is 250 ug/mL or 50 ug/mL depending on which autosampler you will be using. The standard must be prepared every 14 days and stored on the Archon and/or the Centurion autosampler in a pressurized vial or in the VOA standards freezer between uses.

Pentafluorobenzene
1,4-Difluorobenzene
Chlorobenzene-D_5
1,4-Dichlorobenzene-D_4

5.3.2.6 BFB Solution - Prepare a standard as above containing 4-BFB. The final concentration is 25 ug/mL. The standard must be prepared every 30 days and stored in the VOA standards freezer between uses.

5.3.2.7 See Table 4 for a complete list of standards, concentration, and vendors.

NOTE: The concentrations of standards may vary depending on the type of autosampler being used.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

All aqueous samples must be analyzed within 14 days from sample collection if preserved (by addition of HCl to pH <2) or within 7 days from sample collection if unpreserved. All soil/sediments must be analyzed within 14 days from sample collection. For specific projects, soil may be received in pre-weighed vials containing methanol, with an aliquot of the methanol used for analysis. For these projects, the methanol aliquot must be analyzed within 14 days from sample collection. Samples must be stored at $4^{\circ}C \pm 2^{\circ}C$ from the time of receipt at the lab until analysis.

7.0 PROCEDURES

7.1 NAMING AND CODING CONVENTIONS FOR ANALYTICAL STANDARDS – Used in accordance with SOP CA-106 “Standard Preparation and Documentation”.

7.2 COMPUTER (DATA SYSTEM) CONVENTIONS -

Conventions for all instruments are as follows:

Sub-Directory for data acquisition: C:\HPCHEM\1\DATA

Tune file: BFB.U

Method files: I826AXX.M (all samples and standards)
Where:
XX = the calibration number in chronological order
I = instrument ID (C, D, F, M, S or T).
A = matrix (A for water, S for soil and SB for sodium bisulfate soils)

BFB288AQ.M (waters) or BFB288SL.M (soils) (BFB tuning acquisition)

Data files for BFB: IB_ _ _.D where _ _ _ is a number in chronological order from 000 to 999, and I is the instrument ID (C, D, F, M, S or T).

All other data files: I_ _ _ _.D where _ _ _ _ is a number in chronological order from 0000 to 9999, and I is the instrument ID (C, D, F, M, S or T). This file also contains the Quantitation output file.

7.3 INSTRUMENT TUNING - Prior to the analysis of any calibration standards, blanks, or samples, the GC/MS system must be shown to meet the mass spectral ion abundance criteria for a 50 ng injection of p-Bromofluorobenzene (p-BFB), tabulated below:

Mass	Criteria
50	15.0-40.0% of mass 95
75	30.0-60% of mass 95
95	base peak, 100% relative abundance
96	5.0-9.0% of mass 95
173	less than 2.0% of mass 174
174	greater than 50.0% of mass 95
175	5.0-9.0% of mass 174
176	greater than 95.0%, but less than 101.0% of mass 174
177	5.0-9.0% of mass 176

7.3.1 The following are the GC/MS operating conditions for injection of BFB.

GC/MS type: 5970

Column:	RTX-VMS, 40 meter, 0.18 mm ID
Temperatures: Injection port:	170°
Transfer line:	150°
Source:	170°
Analyzer:	170°
Isothermal temperature:	150°
Run time:	10 minutes
Scan start time:	4 minutes
Scan parameters:	not to exceed 2 sec per scan
Mass range:	35-300
Number of A/D samples:	8
GC peak threshold:	1000 counts
Threshold:	10 counts

GC/MS type: 5972 and 5973

Column:	RTX-624, 40 meter, 0.18 mm I.D or RTX-VMS, 40 meter, 0.18 mm ID.
Temperatures: Injection port:	200°
Transfer line:	150°
Detector:	240°
Isothermal temperature:	150°
Run time:	8 minutes
Scan start time:	3 minutes
Scan parameters:	not to exceed 2 sec per scan
Mass range:	35-300
Number of A/D samples:	8
GC peak threshold:	1000 counts
Threshold:	10 counts

The BFB solution must be analyzed once at the beginning of each 12-hour period, the time stamp of the injection of the BFB is the beginning of the 12-hour clock. All calibrations and samples must be run within the 12-hour clock as the method specifies.

When the BFB run has concluded, the run must be evaluated to determine if sample analysis can proceed. The chromatography and the ion ratios must be examined. The BFB run is processed using the current algorithms in the Target software.

If the results indicate the system does not meet acceptance criteria, the GC/MS must be manually tuned. Once the manual tune procedure is completed, BFB must be re-injected and reevaluated. If the instrument still does not meet criteria, notify your Department Manager. Under no circumstances should calibration proceed if the instrument BFB tune is not in criteria.

7.4 INSTRUMENT CONFIGURATION / CALIBRATION

7.4.1 Tekmar LSC 3000/Archon 5100/ Tekmar 2016, Setup/Operation: Please refer to the Tekmar or Archon Manuals for more detailed operations for these instruments.

To begin, set the Tekmar LSC 2000/3000 to the specification listed in section 2-12 of the Archon manual. Edit method 14 as follows:

Method 14 should include:

Standby:	35°
Prepurge:	0 min
Preheat Temp:	0°
Sample Temp:	0°
Purge:	11 min
Dry purge:	2-4 min
Desorb preheat:	245°
Desorb Temp:	250°
Desorb time:	2-5 min
Dry purge:	2-4 min
Bake Time:	10 min
Bake Temp:	260°
Auto drain:	On
Bake gas by pass:	Off
Valve Temp:	120°
Line Temp:	120°
Runs per sample:	1

The above temperature settings are for a Vocarb 3000 trap, these temperatures may vary with the use of alternative traps. Temperature settings may also vary to optimize system performance.

The Archon autosampler should be set up according to the specifications in the manual. The setting of particular concern, with regards to keeping the Tekmar and Archon in coordination with each other, is the desorb time. There are several other programmable features on the Archon; the settings for this feature will depend on the sample matrix and method of analysis. Please refer to the Archon manual for more specifics on its programming features.

7.4.2 Encon/Centurion, Setup/Operation

Please refer to the Encon or Centurion manuals for more detailed operations for the instruments.

To begin, the Encon operation method should contain:

Purge Conditions:	Purge Gas: Helium Purge Time: 11.0 ±0.1 minute Purge Flow Rate: approx. 24-40 mL/min Purge Temperature: Ambient (water)
Desorb Conditions:	DesorbTemp: 250ºC Desorb Flow rate: 15 mL/min Desorb Time: 2.0 ± 0.1 min Bake Time: 10 min Bake Temperature: 260ºC

The above temperature settings are for a Vocarb 3000 trap, these temperatures may vary with the use of alternative traps. Temperature settings may also vary to optimize system performance.

The Centurion autosampler should be set up according to the specifications in the manual.

7.4.3 Initial Calibration for Method 8260

Once the instrument has achieved BFB tuning criteria, calibration of the instrument can begin.

To determine the linearity of response, the GC/MS must be initially calibrated at six different levels.

For aqueous calibration, target analytes and surrogate are prepared at the following concentrations; 1.0, 5.0, 20, 50, 100 and 200 ug/L. The curve is analyzed at ambient temperature.

For a soil calibration target analytes and surrogates are prepped at the following concentrations: 5.0, 10, 20, 50, 100 and 200 ug/L. The calibration standards are stirred and heated to 40ºC.

The following amounts standards should be added to 100 mL of organic-free laboratory reagent grade water in order to generate a 6-point initial calibration curve:

	STD. ID	CAL. Mix 200 ug/mL	Extras Mix 200 ug/mL	Surr. Mix 250 ug/mL Archon	Surr. Mix 50 ug/mL Centurion
AQ curve only	VSTD001	0.5 uL	0.5 uL	0.4 uL	2.0 uL
	VSTD005	2.5 uL	2.5 uL	2.0 uL	10 uL
SL curve only	VSTD010	5.0 uL	5.0 uL	4.0 uL	20 uL
	VSTD020	10 uL	10 uL	8.0 uL	40 uL
CC	VSTD050	25 uL	25 uL	20 uL	100 uL
	VSTD100	50 uL	50 uL	40 uL	200 uL
	VSTD200	100 uL	100 uL	80 uL	400 uL

The internal standard is spiked by the autosampler. Due to different spike amounts separate standards are used depending on which autosampler is being used.

After analysis of the six points, the standard analyses must be quantitated and evaluated for adherence to QC criteria, as follows. Minimum requirements for method files are use of specific quantitation ions and quantitating a specific set of target compound and surrogates with a specified internal standard. These requirements are found in Tables 3 and 5.

7.4.4 Initial Calibration Criteria

The percent (%) RSD for six calibration check compounds (CCC) must be less than or equal to 30%. CCCs are 1,1-Dichloroethene, Chloroform, 1,2-Dichloropropane, Toluene, Ethylbenzene, and Vinyl Chloride.

A system performance check must be performed as part of initial calibration. The five system performance check compounds (SPCC) and the minimum acceptable average relative response factors (RRF) for these compounds are as follows (taken from 8260B):

SPCC	RRF
Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

The SPCCs are used to check both the standard and instrument stability.

7.4.4.1 Linearity of Target Analytes

If the RSD of any target analyte is 15% or less using the average response factor, then the response factor is presumed to be constant

over the calibration range, and the average response factor may be used for quantitation.

If the RSD of any target analyte exceeds 15% using the average response factor, then a calibration option outlined in section 7.0 of method 8000 will need to be employed. Please note that some options may not be allowable for certain states, federal programs, or clients.

Option 1 (Section 7.5.2 of method 8000 - Rev. 2, 12/96), is a linear regression of instrument response versus the standard concentration. The correlation coefficient (r) for each target analyte and surrogate must be greater than or equal to 0.995. For linear models, Target calculates the correlation coefficient and then squares it (r^2). This is what is reported on all Target forms. The value for r^2 must be greater than or equal to 0.990.

Option 2 (Section 7.5.3 of method 8000 - Rev. 2, 12/96), is a non-linear calibration model not to exceed a third order (seven calibration points required) polynomial. The lab would use a quadratic model or second order polynomial. The use of a quadratic model requires six calibration points. In order for the quadratic model to be acceptable, the coefficient of determination must be greater than or equal to 0.99.

7.4.5 Independent Calibration Verification

Immediately following an initial calibration, an independent calibration standard must be analyzed. This standard contains all target compounds, internal standards and surrogates at a concentration of 50 ug/L and is obtained from a source independent of the initial calibration source. Please refer to section 8.1 and Table 1 for acceptance criteria and corrective action for this standard.
For projects or clients requiring DoD QSM 4.1 all project analytes must fall between 80-120% of the true value. No samples may be run until the ICV criteria are met.

7.4.6 Calibration Verification

Once a valid initial calibration curve has been achieved, a continuing calibration standard containing all the target compounds, internal standards and surrogates at a concentration of 50 ppb must be analyzed every 12-hour clock for Method 8260, timed from the injection of BFB. The relative response factor from the 50 ppb continuing calibration check standard must be compared to the average response factor data from the initial calibration.

The EICP (extracted ion current profile) area for any of the internal standards in the calibration verification must not change by more than a factor of two (-50% to +100%) from the same level standard in the last initial calibration. The retention time for any internal standard cannot shift by more than 30 seconds from the same level standard in the last initial calibration.

For Method 8260, if the percent difference for each CCC is less than or equal to 20%, and all of the SPCCs have a relative response factor greater than or equal to those listed in Section 7.4.3, the continuing calibration is considered valid.

For projects or clients requiring DoD QSM 4.1 all project analytes must have ± 20%D.

Continuing calibration check criteria must be met before sample analysis can proceed.

7.4.7 Retention Time Windows

Retention time windows are set at the midpoint standard of the calibration curve, following every ICAL. When a CV is analyzed (and not an ICAL), the retention time windows of the daily CV must be within 30 seconds of the midpoint calibration standard of the most recent ICAL. The samples analyzed following the daily CV must have retention times within 30 seconds of those for the daily CV. Each successive daily CV must be compared to the most recent ICAL midpoint standard.

For projects or clients requiring DoD QSM 4.1, IS responses and retention time windows for QC and samples are compared to the midpoint of the most recent ICAL.

7.5 QUALITY CONTROL SAMPLE ANALYSIS

When preparing standards in water or spiking samples with internal standards/surrogates or matrix spike solution, be sure to rinse all syringes a minimum of three times with purge and trap grade methanol between uses. Failure to do this will result in cross-contamination of samples and standards.

7.5.1 Laboratory Control Sample (LCS)

The LCS mix is prepared from a secondary source vendor (i.e. different vendor from the calibration standards). The LCS is analyzed immediately after the initial calibration curve or calibration check and prior to the method blank to minimize any analyte carryover possibilities in samples. Acceptance criteria for the LCS are outlined in Section 8.0.

To prepare the water and medium-level soil LCS, 25 uL of the LCS standard mix at 200 ug/mL are spiked into 100 mL of analyte-free laboratory reagent grade water for a final concentration of 50 ug/L. The Archon autosampler adds 1 uL of internal and 1 uL of surrogate standard to a 5 mL aliquot of this preparation for analysis. The Centurion autosampler adds 5 uL of both surrogates and internal standards to a 5 mL aliquot. To prepare the low-level soil LCS, a stir bar is added to 5 mL of the above solution in a VOA vial. The Archon unit adds an additional 10 mL of water to which the internal and surrogate standards have been added; this preparation is then heated, stirred and purged.

To prepare the water and medium-level soil LCS for analysis on the LSC 2000 / 2016 autosampler, 1.25 uL of the LCS standard mix at 200 ug/mL are spiked into 5 mL of analyte-free laboratory reagent grade water for a final concentration of 50 ug/L.

In the event that the batch MS/MSD requirement cannot be fulfilled, a laboratory Control Spike Duplicate must be analyzed.

7.5.2 Method Blank Analysis

After calibration criteria have been met, a method blank must be analyzed before sample analysis can proceed. A method blank analysis must be performed once for each 12-hour calibration immediately after analysis of the calibration standard(s) and prior to sample analysis.

The aqueous method blank is a volume of analyte free laboratory reagent grade water spiked with internal and surrogate standards.

The low-level soil method blank is a volume of analyte free laboratory reagent grade water spiked with internal and surrogate standards. This method blank is analyzed using the low soil specification.

The method blank must contain less than the Practical Quantitation Level (PQL) for all analytes of interest for the samples associated with the blank.

For projects requiring DoD QSM 4.1 no analytes may be detected >1/2 the PQL and > than the $1/10^{th}$ the measured amount in any sample or $1/10^{th}$ the regulatory limit, whichever is larger. Except for common laboratory contaminants which may not be detected > than the PQL.

7.5.3 Surrogate Recovery Limits

Laboratory established limits are derived for each of the surrogates. Please refer to the current revision of Katahdin Analytical Services SOP # QA-808 for further information on statistical limits. All samples including blanks, laboratory

control samples, matrix spikes and client samples, must meet the statistical limits for the analysis to be considered valid. If surrogate recoveries do not meet these limits, reanalysis must occur to confirm matrix interference.

7.5.4 Internal Standard Area Recoveries / Retention Times.

The internal standard responses and retention times in the method blank must be evaluated immediately after or during data acquisition. If the EICP (extracted ion current profile) area for any of the internal standard changes by a factor of two (-50% to +100%), from the last daily calibration standard, the GC/MS must by inspected, and corrective action taken. If the retention time for any internal standard has shifted by more than 30 seconds from the mid-point standard level of the most recent calibration sequence, the GC/MS must be inspected, and corrective action taken. All samples and QC must also meet the EICP area and retention time criteria or must be reanalyzed.

7.5.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis

An MS/MSD must be analyzed every twenty samples of a similar matrix. The MS/MSD is prepared in a manner similar to the LCS, except that 40 mL aliquots (aqueous) or 5 g aliquots (soil), of environmental samples are used in place of the analyte-free laboratory reagent grade water. Note that trip blanks and field/equipment blanks should not be used for MS/MSD analyses. The spike solution (section 7.5.1) is added to the sample at a concentration of 50 ppb. Acceptance criteria for the MS/MSD are outlined in Section 8.0.

In the event that sufficient volume of sample is not supplied to the laboratory so that an MS/MSD set cannot be analyzed within a batch of 20 samples, a laboratory control spike duplicate must be analyzed.

7.6 SAMPLE ANALYSIS

When new samples are received, they should be checked for past sample history. If sample history cannot be located or the sites are different than past sites, the project manager should be consulted. He/she may be able to provide more information about the sample. Sample history is used to determine what order in which to run the samples and at what dilution. Refer to Katahdin Analytical Services SOPCA-106, “Basic Laboratory Technique”, current revision for information on subsampling.

Samples are removed from the VOA refrigerator and appropriate chain of custody form is completed. Remove only the vials that have not been opened yet (opened vials will be upside down). Note in sample run log any bubbles, and significant discoloration or sediment in the sample vials.

7.6.1 SAMPLE ANALYSIS FOR 8260B WATER

7.6.1.1 Tekmar LSC 2000 / 2016 units

Rinse a 5.0 mL gas-tight syringe a minimum of three times with analyte-free laboratory reagent grade water (e.g., Poland Spring or equivalent). Pour sample at ambient temperature into the syringe until nearly overflowing. Carefully insert and adjust plunger to sample volume of 5.0 mL. While adjusting plunger to final volume, expel extra volume of sample onto pH paper for sample pH verification. Add 1.0 uL of the internal and surrogate mixtures (250 ug/mL). Immediately inject contents of the syringe into the ALS sparger.

Record the sample pH in the injection logbook. Continue as above for each sample, ensuring that the 5.0 mL gas-tight syringe is rinsed a minimum of three times with laboratory reagent grade water between each sample.

7.6.1.2 Tekmar LSC 3000 / Archon 5100 units

Place the sample vials into the Archon sample tray and program the Archon for the appropriate sample volume and or dilution for the sample. The Archon unit will automatically transfer the sample to the sparge vessel while adding the internal and surrogate standard. The Archon can be programmed to run as many samples as will fit in the twelve-hour window. The auto sampler hot water rinses the sparge vessel, transfer lines, purge needle, and syringe between samples to minimize possible carryover.

Record the sample pH in the injection logbook after sample analysis is complete (usually the day after the analysis is done) and return the sample vial to the sample refrigerator.

7.6.1.3 Centurion/Encon unit

Place the sample vials into the Centurion sample tray and program the Centurion for the proper sequence. The Centurion will automatically transfer the sample to the sparge vessel while adding the internal and surrogate standards. Using the Centurion software, the analyst can program the Centurion to run as many samples that will fit into a 12 hour clock. The autosampler uses hot water to rinse the sparge vessel, transfer lines, purge needle and sample needle to minimize carryover.

Record the sample pH in the injection logbook after sample analysis is complete (usually the day after the analysis is done) and return the sample vial to the sample refrigerator.

Make sure that all entries in the injection log have been made in a complete, neat, and legible manner. Corrections in any logbook must be crossed through with a single line, dated, initialed and have a written explanation or the applicable error code.

If for any reason a sample needs to be rerun, diluted or duplicated, a note in the comments field of the injection logbook must be entered, addressing the reason why in the logbook to facilitate answering any questions that may arise during the review process.

To minimize carryover from samples that contain a target compound at a level exceeding the upper limit of the calibration curve, the following must be done: monitor both the samples immediately after the contaminated sample as well as the next run of the contaminated sample in the same purge inlet for the target(s) in question; both must have levels <PQL.

7.6.2 ANALYSIS OF LOW-LEVEL SOIL SAMPLES

Method 5035 Closed System Purge & Trap procedure for low level soils (5 ug/Kg -200 ug/Kg)

Selecting the appropriate technique may depend on cleanup goals, confidence levels, and anticipated levels of contamination. Field sampling activities typically result in Encore or Encore-like devices being submitted to the lab. These devices must be extruded within 48 hours. It is the laboratory's standard policy to extrude soil samples into 5 mL of Laboratory reagent free laboratory reagent grade water that contains a magnetic stir bar. The sample is subsequently frozen until analysis within 14 days. Note that the sample must be extruded and frozen within 48 hours of sampling, until analysis can begin. This approach is preferred over extrusion into sodium bisulfate because it is believed that the sodium bisulfate reacts with calcium carbonate in highly calcareous soils causing effervescence and driving the volatile analytes out of solution. There is also anecdotal information to suggest that acetone may be generated when bisulfate preservation occurs. The Katahdin sample ID, extrusion date, and time are recorded in the GC/MS extrusion logbook. Please refer to the Katahdin method 5035 SOP, CA-214 for more detail.

In lieu of the use of Encore samplers, the lab may pre-weigh 40 mL VOA vials containing 5 mL of laboratory reagent grade water or a 20% sodium bisulfate solution and a magnetic stir bar and ship these to the field. The vial

is assigned a vial specific number prior to shipment to the field. The vial and weight will be recorded with its vial specific number in the methanol soil logbook. If possible the field sampler should weigh the sealed vial to ensure that 5 +/- 0.5 grams of sample were added in the field. When the lab receives the vials back from the field, the vials will be weighed and the weight recorded. The samples must be frozen within 48 hours of sampling, until analysis can begin.

The subsequent analysis is performed on a specially developed autosampler that heats, stirs, and purges the sample simultaneously without exposing the contents of the vial to the atmosphere. This procedure will help to minimize the loss of VOC's due to transport, handling, and analysis and may help minimize ambient lab contribution. The expected detection limits are consistent with the traditional low soil technique from method 5030. The Archon is programmed to heat each vial to 40°C during the purge time. Initiate purging for 11.0 minutes; the sample must be heated to 40°C ± 1°C before purging can begin. If you have questions concerning setting up the Tekmar or initiating a GC/MS batch run, consult the Organic Department Manager, or senior chemist within the group.

If the client does not require method 5035, method 5030 for analysis of low-level soils may be followed. This means that the Tekmar ALS 2016 unit may be used for the preparative step, as well as the Archon units.

7.6.3 ANALYSIS OF MEDIUM-LEVEL SOIL SAMPLES

Method 5030 Procedure for higher concentration soils (> 200 ug/Kg)

Higher concentration soils may be sampled as either a bulk sample or field preserved with a water miscible solvent such as methanol. If sampled in an Encore unit, the soil is extruded into methanol upon receipt at the lab.

Bulk Sample- A sample is placed in a glass jar or vial and returned to the lab for extraction and analysis. In this approach the lab takes an aliquot of soil and extracts with purge & trap grade methanol, a portion of the methanol is then analyzed for volatile analytes.

Extraction

Calibrate the balance properly (See SOP CA-102) and note it in the appropriate logbook. Place 5.0 grams of thoroughly mixed, undecanted soil sample in a 40.0 mL vial. Add 5.0 mL reagent grade methanol. Shake for 2 minutes. Let stand for 3 minutes. Record extraction in soil prep logbook.

Methanol Field Preservation - A 5 gram sample is added to a VOA vial that has been previously charged with purge and trap grade methanol (the

volume of methanol is dependent upon client request). The vial with methanol has been previously weighed in the lab and assigned a vial specific number prior to shipment to the field. The vial and methanol weight will be recorded with its vial specific number in the VOA vial prep logbook. If possible the field sampler should weigh the sealed vial to ensure that 5 +/- 0.5 grams of sample were added in the field. When the lab receives the vials back from the field, the vials will be weighed and the weight recorded. A portion of the methanol is then analyzed for volatile analytes.

For analysis on Archon or Centurion autosamplers, add 400 uL of the extract into 20 mL of organic-free laboratory reagent grade water (e.g., Poland Spring or equivalent). IS and SS is added by the Archon and/or Centurion autosampler for analysis. This will give an estimated calibration range between 500-10000 ug/Kg.

7.7 FINAL DATA PACKAGE

7.7.1 Initial Data Review (IDR)

The initial data review is performed by the analyst who ran the samples. This review is of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed. The initial data review is performed on the detailed quantitation reports of the analyzed sample. This data review examines criteria that directly impact whether or not the sample needs to be reanalyzed.

- Surrogate recoveries
- stability of internal standard responses
- LCS spike recoveries
- method blank acceptance
- chromatography
- target compound detection/quantitation / review for false positives

The analyst must evaluate all data using the QA Acceptance Criteria table found within this SOP (Table 1). This table gives acceptance criteria and corrective actions for criteria that are not met. In addition to evaluating QC elements, the chromatography and quantitation of target analytes must be reviewed.

7.7.1.1 Chromatography

The chromatography should be examined for the presence or absence of any "ghost" peaks and can also be used as an indication of whether or not matrix interferences might be influencing surrogate recoveries and/or ISTD area recoveries. Whether or not the chromatography is

acceptable is a judgment call on the part of the analyst and should be used in conjunction with other monitored QC (e.g., Surrogate recoveries) to determine the necessity of reanalyses.

Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating the peak of concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.

Each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. An "M" qualifier will automatically be printed on the quantitation report summary.
This manual integration package must then be submitted to the Organic Department Manager or his/her designee, who will review each manual integration.

For specific procedures on how to manually integrate, refer to Katahdin SOP QA-812, "Manual Integration", current revision.

7.7.1.2 Target Compound Detection/Quantitation

The method files have been set up to error on the side of false positives, that is to identify and quantitate peaks as target compounds that may not necessarily be valid hits.

The requirements for qualitative verification by comparison of mass spectra are as follows:

- all ions present in the standard mass spectra at a relative intensity > 25% must be present in the sample spectrum.
- the relative intensities of primary and secondary ions must agree within ±20% between the standard and sample spectra.
- ions greater than 25% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst.

If a compound cannot be verified by all three criteria above, but, in the technical judgment of the mass spectral interpretation specialist, the identification is correct, then the laboratory shall report that compound on the Form 1 as a valid hit.

If any target concentration exceeds the upper limit, a dilution must be made and analyzed. The dilution chosen should keep the response of the largest target compound hit in the upper half of the initial calibration range.

The GC/MS laboratory initial data review must be completed within twelve hours of batch completion; in the majority of instances, the initial data review should be accomplished at the beginning of a work shift for the previous set of analyses. After the analyst has completed his or her initial data review, the data should immediately be forwarded to the Organic Department Manager, or his/her designee.

7.7.1.3 Tentatively Identified Compounds (TIC)

TIC's may be requested by certain clients for samples. Refer to SOP CA-207 "GC/MS Library Search and Quantitation".

7.7.2 Reporting

After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into QuickForms. Depending on the QC level requested by the client, a Report of Analysis (ROA) and additional reports, such as LCS forms and chronology forms, are generated. The package is assembled to include the necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded to the secondary reviewer. The secondary reviewer validates the data and checks the package for any errors. When completed, the package is sent to the department manager for final review. A completed review checklist is provided with each package. The final data package from the Organics department is then processed by the Data Management department.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 and to details in this section for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The criteria does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in this section or in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in this section and in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The Department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work

performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 Independent Calibration Verification, LCS and MS/MSD Criteria

Statistical limits are compiled annually for LCS recoveries (archived in QA office). Statistical limits are only calculated when at least 30 usable data points are obtained for any given compound. If insufficient data points are available, nominal limits are set by the Organic Department Manager, Laboratory Operations Manager and Quality Assurance Officer. Refer to Katahdin SOP QA-808, "Generation and Implementation of Statistical QC Limits and/or Control Charts," current revision.

The use of statistical limits versus nominal limits is dependent on the client and project. This information is communicated to the Organic Department Manager through the Katahdin project manager. It is standard practice to use statistical limits for reporting purposes and to evaluate any QC criteria exceedances. However, nominal limits of 60-140% or 70-130% may be used for some projects or states.

The LCS recoveries for all analytes are evaluated. All of the compounds of interest must fall within the established statistical limits with the following sporadic exceedance allowances.

Number of Analytes	Number of Allowable Exceedances
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
<11	0

If less than the number of allowable exceedances fail the statistical limits, no corrective action is needed. If greater than the number of allowable exceedances fail the statistical limits, corrective action may be taken. Corrective actions may vary with each situation. However, in the case where the failures are high and the samples are non-detect for those compounds, then no corrective action is required. Otherwise, corrective action may involve reanalysis or recalibration. The specific corrective actions taken will rely on analyst experience to make sound scientific judgments while considering client objectives, other quality control indicators and/or the ability to reanalyze a sample within holding time.

The MS/MSD recoveries for all analytes are evaluated. If the LCS results are acceptable but the MS/MSD is not, narrate. If both the LCS and MS/MSD are unacceptable reprep the samples and QC.

Please note that for compounds with only nominal limits (i.e. insufficient data points were available to generate statistical limits), no corrective action is required for out-of-criteria recoveries until enough data points are established to generate statistical limits.

For projects or clients requiring DoD QSM 4.1 all project analytes in the ICV must fall between 80-120% of the true value. No samples may be run until the ICV criteria is met. Laboratory established recovery limits for LCS and MS/MSDs must be within 3 standard deviations of the mean LCS recovery. MS/MSD pairs must be run once per analytical/preparatory batch. RPDs must be less than or equal to 30% between MS and MSDs.

For analytes with no available DoD acceptance criteria, laboratory established limits shall be used.

8.2 Surrogate Recovery Criteria

Statistical limits are compiled annually for surrogate recoveries (archived in QA office). Statistical limits are only calculated when at least 30 usable data points are obtained for any given compound. If insufficient data points are available, nominal limits are set by the Organic Department Manager, Laboratory Operations Manager and Quality Assurance Officer. The use of statistical limits versus nominal limits is dependent on the client and project. This information is communicated to the Organic Department Manager through the Katahdin project manager. It is standard practice to use statistical limits for reporting purposes and to evaluate any QC criteria exceedances. However, nominal limits of 60-140% or 70-130% may be used for some projects or states.

8.3 QC Requirements

Refer to Table 1 for a summary of QC requirements, acceptance criteria, and corrective actions. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The Department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Due to the 14-day hold time associated with this method, samples may not be able to be reanalyzed

within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Organic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revision of Method 8260 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007, Method 8260B.

"Department of Defense Quality Systems Manual for Environmental Laboratories" (DoD QSM), Version 4.1, 04/22/09.

"The National Environmental Laboratory Accreditation Conference (NELAC) Standards," June 2003.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

LIST OF TABLES AND FIGURES

TABLE 1

QC REQUIREMENTS - VOLATILE ORGANICS, METHOD 8260

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Check of mass spectral ion intensities using BFB	Prior to initial calibration and calibration verification	Refer to the criteria listed in Section 7.3 of this SOP	Retune instrument, and verify
Six-point calibration for all analytes	Initial calibration prior to sample analysis	SPCCs average RF ≥0.30, except chloromethane, 1,1-DCA and bromoform ≥0.10; RSD for RFs ≤ 30% for CCCs. Refer to section 7.4.3 also.	Repeat initial calibration
Independent Calibration Verification	Once, immediately following calibration	Statistically derived from lab data or nominal limits depending on the project. Refer to QA records for statistical limits. Nominal limits are used as default limits. See also section 8.1 of this SOP for more information on allowable exceedances	Evaluate the samples and associated QC: i.e. If an MS/MSD was performed and acceptable, narrate. If an LCS/LCSD was performed and only one of the set was unacceptable, narrate. If the surrogate recoveries in the LCS are also low but are acceptable in the blank and samples, narrate. If the LCS recovery is high but the sample results are <PQL, narrate. Otherwise, reprep a blank and the remaining samples.
Calibration verification	Once per each 12 hours, prior to sample analysis in absence of initial cal	SPCCs minimum RF ≥ 0.30, except chloromethane, 1,1-DCA and bromoform ≥ 0.10; RF for CCC analytes ≤ 20% (%D) of average initial multipoint RF	Repeat initial calibration and reanalyze all samples analyzed since the last successful calibration verification
IS	During data acquisition of calibration check standard	Retention time ± 30 seconds; EICP area within -50% to +100% of last calibration verification (12 hours) for each IS	Inspect mass spectrometer or GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning
Method Blank	One per batch of 20 or fewer samples.	No analytes of interest detected > PQL with the exception of Methylene Chloride	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e. If the blank results are above the PQL, report sample results which are <PQL or > 10X the blank concentration. Otherwise, reprep a blank and the remaining samples.

TABLE 1 (cont.)

QC REQUIREMENTS - VOLATILE ORGANICS, METHOD 8260

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
LCS	One per batch of 20 or fewer samples.	Statistically derived from lab data or nominal limits depending on the project. Refer to QA records for statistical limits. Nominal limits are used as default limits. See also section 8.4 of this SOP for more information on allowable exceedances. .	Evaluate the samples and associated QC: i.e. If an MS/MSD was performed and acceptable, narrate. If an LCS/LCSD was performed and only one of the set was unacceptable, narrate. If the surrogate recoveries in the LCS are also low but are acceptable in the blank and samples, narrate. If the LCS recovery is high but the sample results are <PQL, narrate. Otherwise, reprep a blank and the remaining samples.
Surrogate spike	Every sample, control, standard and method blank	Statistically derived limits.	Reprep and reanalyze for confirmation of matrix interference when appropriate.
MS/MSD	One MS/MSD per every 20 samples.	Statistically derived from lab data or nominal limits depending on the project. Statistical limits are used as default limits. .	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS/MSD are unacceptable reprep the samples and QC.
MDL Study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		
Demonstrate ability to generate acceptable P & A using 4 replicate analyses of a QC check standard	Once per year for each analyst; 4 reps	All recoveries within method QC acceptance limits	Recalculate results; locate and fix problem; rerun P & A study for those analytes that did not meet criteria prior to sample analysis

TITLE: **ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

TABLE 2

DOD QSM Version 4.1 QC Requirements

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specific criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria.	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification	Refer to current revision of SOP QA-806				
LOQ establishment and verification	Refer to current revision of SOP QA-806				
Tuning	Prior to ICAL and at the beginning of each 12-hour period.	Refer to method for specific ion criteria.	Retune instrument and verify. Rerun affected samples.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be accepted without a valid tune.
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	1. Average response factor (RF) for SPCCs: VOCs ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachlorolethane; ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. 2. RSD for RFs for CCCs ≤ 30% and one option below: Option 1: RSD for each analyte ≤ 15%; Option 2: linear least squares regression r ≥ 0.995; Option 3: non-linear regression–coefficient of determination (COD) r2 ≥ 0.99 (6 points shall be used for second order).	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. Calibration may not be forced through the origin.
Second source calibration verification (ICV)	Once after each ICAL.	All project analytes within ± 20% of true value.	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.

TITLE: **ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

TABLE 2 (cont)

DOD QSM Version 4.1 QC Requirements

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Retention time window position establishment for each analyte and surrogate	Once per ICAL.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	
Evaluation of relative retention times (RRT)	With each sample.	RRT of each target analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	Flagging criteria are not appropriate.	Laboratories may update the retention times based on the CCV to account for minor performance fluctuations or after routine system maintenance (such as column clipping). With each sample, the RRT shall be compared with the most recently updated RRT. If the RRT has changed by more than ±0.06 RRT units since the last update, this indicates a significant change in system performance and the laboratory must take appropriate corrective actions as required by the method and rerun the ICAL to reestablish the retention times.
Continuing calibration verification (CCV)	Daily before sample analysis and every 12 hours of analysis time.	1. Average RF for SPCCs ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachlorolethane; ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. 2. %Difference/Drift for all target compounds and surrogates ≤ 20%D (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration).	DoD project level approval must be obtained for each of the failed analytes or corrective action must be taken. Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since last acceptable CCV.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Internal standards verification	Every field sample, standard, and QC sample.	Retention time ± 30 seconds from retention time of the midpoint standard in the ICAL; EICP area within -50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, apply Q-flag to analytes associated with the non-compliant IS. Flagging criteria are not appropriate for failed standards.	Sample results are not acceptable without a valid IS verification.

TABLE 2 (cont)

DOD QSM Version 4.1 QC Requirements

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method blank	One per preparatory batch.	No analytes detected > ½ RL (> RL for common lab contaminants) and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Reprepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result. Contact Client if samples cannot be reprepped within hold time.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
LCS containing all analytes to be reported, including surrogates	One per preparatory batch.	The laboratory shall use laboratory control limits (CLs) or use DoD-generated LCS-CLs, if available depending on project requirements. In-house CLs may not be greater than ± 3 times the standard deviation of the mean LCS recovery. A number of analytes may fall outside the CL but within marginal exceedance limit depending on the total number of analytes in the LCS.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. Refer to Table G-1 for number of marginal exceedences allowed. Contact Client if samples cannot be reprepped within hold time.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch per matrix if sufficient sample is available.	For matrix evaluation, use LCS acceptance criteria.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix if sufficient sample is available.	MSD: For matrix evaluation, use LCS acceptance criteria. MS/MSD: RPD ≤ 30% .	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.

TABLE 2 (cont)

DOD QSM Version 4.1 QC Requirements

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Surrogate spike	All field and QC samples.	The laboratory shall use laboratory surrogate CLs or use DoD-generated surrogate CLs, if available depending on project requirements. .	For QC and field samples, correct problem then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary. Contact Client if samples cannot be reprepped within hold time.	Apply Q-flag to all associated analytes if acceptance criteria are not met.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

TITLE: **ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

TABLE 3

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-202-11	METHOD 8260, current revision
Apparatus/Materials	None	
Reagents	None	
Sample preservation/ handling	Preserved samples analyzed within 14 days. Unpreserved samples analyzed within 7 days.	Preserved samples analyzed within 14 days. No criteria for unpreserved samples.
Procedures	(1) Use laboratory reagent grade water for low level soil calibration, method blanks, and laboratory control samples to minimize clogging of archon soil needles with sand. (2) Internal Standards- pentafluoro-benzene, 1,4-difluorobenzene, chlorobenzene-d5, 1,4-dichloro-benzene-d4	(1) Use an aliquot of a clean (control) matrix similar to the sample matrix. (2) Recommended internal standards – fluorobenzene, chlorobenzene-d5, 1,4-dichloro-benzene-d4
QC - Spikes	None	
QC - LCS	None	
QC - Accuracy/Precision	PQL – Practical Quantitation Level – three to ten times the MDL.	EQL – Estimated Quantitation Level – five to ten times the MDL
QC - MDL	None	

TABLE 4

VOA COMPOUNDS AND CHARACTERISTIC IONS

COMPOUND	1° ION	2° ION
Acetone	43	58
Acetonitrile	41	40, 39
Acrolein	56	55, 58
Acrylonitrile	53	52, 51
Allyl Chloride	76	41, 39
Benzene	78	-
Bromobenzene	156	77, 158
Bromochloromethane	128	49, 130
Bromodichloromethane	83	85, 127
Bromoform	173	175, 254
Bromomethane	94	96
2-Butanone	43	72
n-Butylbenzene	91	92, 134
Sec-Butylbenzene	105	134
Tert-Butylbenzene	119	91, 134
Carbon Disulfide	76	78
Carbon Tetrachloride	117	119
Chlorobenzene	112	77, 114
Chloroethane	64	66
2-Chloroethylvinyl Ether	63	65, 106
Chloroform	83	85
Chloromethane	50	52
Chloroprene	53	88, 90
2-Chlorotoluene	91	126
4-Chlorotoluene	91	126
Cyclohexane	56	84, 60
1,2-Dibromo-3-Chloropropane	75	155, 157
Dibromochloromethane	129	127
1,2-Dibromoethane	107	109, 188
Dibromomethane	93	95, 174
Diethyl Ether	74	45, 59
1,2-Dichlorobenzene	146	111, 148
1,3-Dichlorobenzene	146	111, 148
1,4-Dichlorobenzene	146	111, 148
Dichlorodifluoromethane	85	87
1,1-Dichloroethane	63	65, 83
1,2-Dichloroethane	62	98
1,1-Dichloroethene	96	61, 63
Cis-1,2-Dichloroethene	96	61, 98
Trans-1,2-Dichloroethene	96	61, 98
1,2-Dichloropropane	63	112

TABLE 4 (cont.)

VOA COMPOUNDS AND CHARACTERISTIC IONS

COMPOUND	1° ION	2° ION
1,3-Dichloropropane	76	78
2,2-Dichloropropane	77	97
1,1-Dichloropropene	75	110, 77
Cis-1,3-Dichloropropene	75	77, 39
Trans-1,3-Dichloropropene	75	77, 39
Cis-1,4-Dichloro-2-butene	75	53, 77
Trans-1,4-Dichloro-2-butene	53	88, 75
1,4-Dioxane	88	58, 43
Di-Isopropyl Ether	45	43, 87
Ethylbezene	91	106
Ethyl Methacrylate	69	41, 99
Ethyl Tertiary-Butyl Ether	59	87, 57
Freon-113	151	101
Hexachlorobutadiene	225	223, 227
2-Hexanone	43	58, 57, 100
Idomethane	142	127, 141
Isobutyl Alcohol	43	41, 42
Isopropylbezene	105	120
P-ISOPROPYLTOLUENE	119	134, 91
Methacrylonitrile	41	67, 39
Methylcyclohexane	83	55, 98
Methylene Chloride	84	86, 49
Methyl Acetate	43	74
Methyl Methacrylate	69	41, 100
4-Methyl-2-Pentanone	43	58, 85, 100
Methyl Tert-Butyl Ether	73	57, 41
Naphthalene	128	-
Pentachloroethane	167	130, 132
Propionitrile	54	52, 55
N-PROPYLBENZENE	91	120
Styrene	104	78
Tertiary-Amyl Methyl Ether	73	55, 87, 71
Tertiary-Butyl Alcohol	59	41, 43
1,1,1,2-Tetrachloroethane	131	133, 119
1,1,2,2-Tetrachloroethane	83	131, 85
Tetrachloroethene	164	129, 131, 166
Tetrahydrofuran	42	72, 71
Toluene	92	91
1,2,3-Trichlorobenzene	180	182, 145
1,2,4-Trichlorobenzene	180	182, 145
1,3,5-Trichlorobenzene	180	182, 145
1,1,1-Trichloroethane	97	99, 61
1,1,2-Trichloroethane	83	97, 85
Trichloroethene	95	97, 130, 132

TABLE 4 (cont.)

VOA COMPOUNDS AND CHARACTERISTIC IONS

COMPOUND	1° ION	2° ION
Trichlorofluoromethane	151	101, 153
1,2,3-Trichloropropane	75	77
1,2,3-Trimethylbenzene	105	120
1,2,4-Trimethylbenzene	105	120
1,3,5-Trimethylbenzene	105	120
Vinyl Acetate	43	86
Vinyl Chloride	62	64
Xylenes (Total)	106	91
1-Chlorohexane	91	55,43

TITLE: **ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

TABLE 5

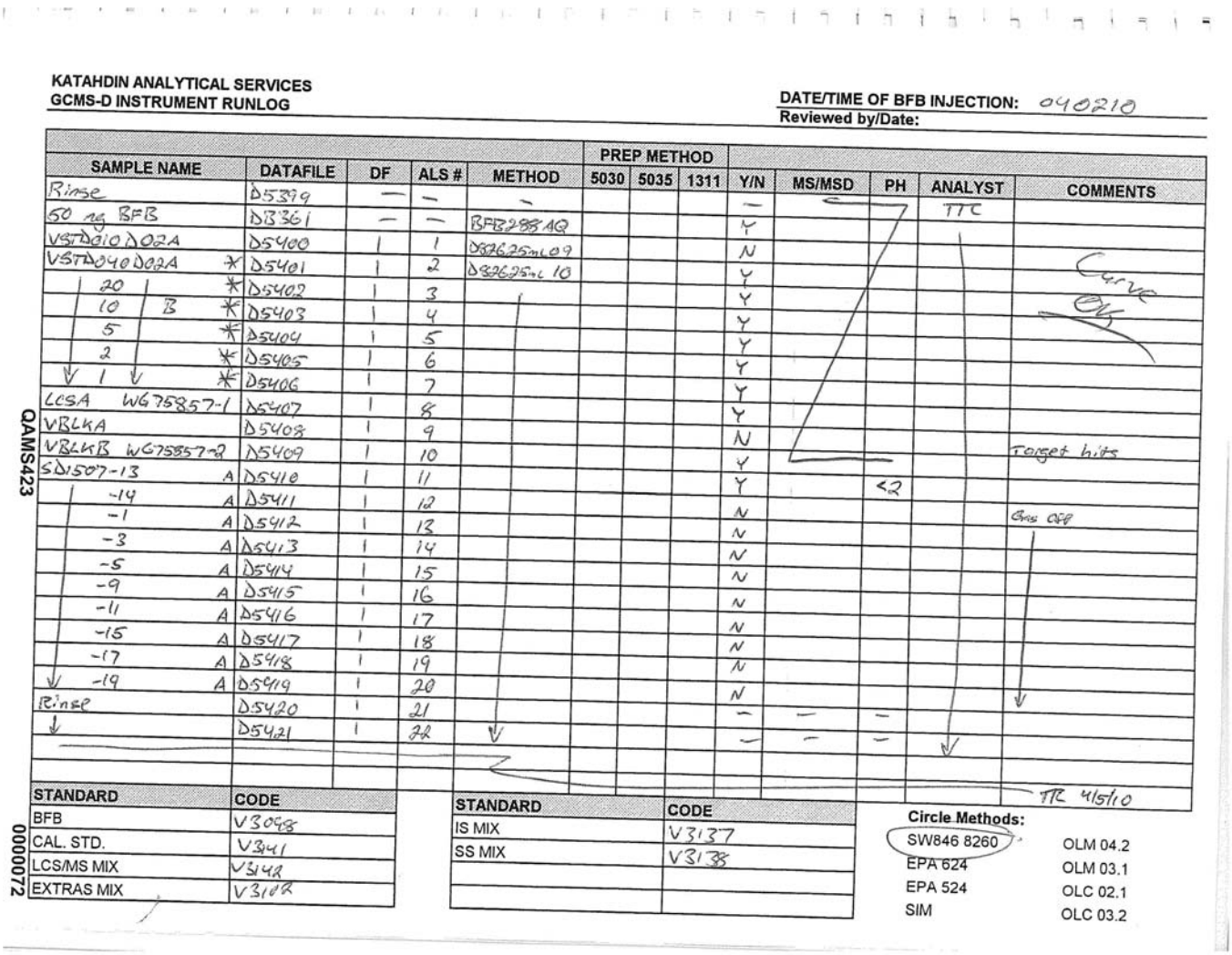
ANALYTE QUANTITATION AND INTERNAL STANDARDS

Pentafluorobenze	**1,4-Difluorobenzene**	**Chlorobenzene - d5**	**1,4-Dichlorobenzene - d4**
Dichlorodifluoromethane	1,2-Dichloroethane	1,3-Dichloropropane	1,1,2,2-Tetrachloroethane
Chloromethane	1,1-Dichloropropene	Tetrachloroethene	1,2,3-Trichloropropane
Bromomethane	Carbon tetrachloride	Dibromochloromethane	Isopropylbenzene
Vinyl chloride	Benzene	Chlorobenzene	Bromobenzene
Chloroethane	1,2-Dichloropropane	1,1,1,2-Tetrachloroethane	2-Chlorotoluene
Trichlorofluoromethane	Trichloroethene	Ethylbenzene	4-Chlorotoluene
Methylene Chloride	Dibromomethane	Xylenes (total)	1,3,5-Trimethylbenzene
Acetone	Bromodichloromethane	Bromoform	Tert-Butylbenzene
1,1-Dichloroethene	cis -1,3-Dichloropropene	Styrene	1,2,4-Trimethylbenzene
1,1-Dichloroethane	4-Methyl-2-pentanone	2-Hexanone	Sec-Butylbenzene
cis-1,2-Dichloroethene	Toluene-d8 (surr.)	Bromoform	1,3-Dichlorobenzene
trans-1,2-Dichloroethene	Toluene		P-Isopropyltoluene
Chloroform	trans-1,3-Dichloropropene		1,4-Dichlorobenzene
2,2-Dichloropropane	1,1,2-Trichloroethane		1,2-Dichlorobenzene
2-Butanone	1,2-Dibromoethane		N-Propylbenzene
Methyl-tert-butylether (MTBE)	Vinyl Acetate		1,2-Dibromo-3-chloropropane
Tetrahydrofuran	Methyl Methacrylate		1,2,4-Trichlorobenzene
Bromochloromethane	Ethyl Methacrylate		Naphthalene
1,1,1-Trichloroethane	1,4-Dioxane		Hexachlorobutadiene
Tertiary-butyl alcohol (TBA)	2-Chloroethylvinyl ether		1,2,3-Trichlorobenzene
Di-isopropyl ether (DIPE)	Bromofluorobenzene (surr.)		cis-1,4-Dichloro-2-butene
Ethyl-tert-butylether (ETBE)			trans-1,4-Dichloro-2-butene
Tertiary-amyl methyl ether			Pentachloroethane
Diethyl Ether			n-Butylbenzene
Carbon Disulfide			1,3,5-Trichlorobenzene
Freon-113			1,2,3-Trimethylbenzene
Iodomethane			
Acrolein			
Isobutyl Alcohol			
Allyl Chloride			
Chloroprene			
Propionitrile			
Methacrylonitrile			
Acrylonitrile			
Cyclohexane			
Methyl Acetate			
Methylcyclohexane			
1-Chlorohexane			
Dibromofluoromethane (surr.)			
1,2-Dichloroethane-d4 (surr.)			

TITLE: **ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

FIGURE 1

EXAMPLE OF VOA RUNLOG PAGE



KATAHDIN ANALYTICAL SERVICES
GCMS-D INSTRUMENT RUNLOG

DATE/TIME OF BFB INJECTION: 040210
Reviewed by/Date:

QAMS423

SAMPLE NAME	DATAFILE	DF	ALS #	METHOD	PREP METHOD 5030	5035	1311	Y/N	MS/MSD	PH	ANALYST	COMMENTS
Rinse	D5399	—	—	—				—			TTC	
50 ng BFB	D3361	—	—	BFB288AQ				Y				
VSTD010D02A	D5400	1	1	D826251L09				N				
VSTD040D02A *	D5401	1	2	D826251L10				Y				
20 *	D5402	1	3					Y				
10 B *	D5403	1	4					Y				
5 *	D5404	1	5					Y				
2 *	D5405	1	6					Y				
1 *	D5406	1	7					Y				
LCSA WG75857-1	D5407	1	8					Y				
VBLKA	D5408	1	9					N				
VBLKB WG75857-2	D5409	1	10					Y				Target hits
SD1507-13 A	D5410	1	11					Y		<2		
-14 A	D5411	1	12					N				
-1 A	D5412	1	13					N				Gas off
-3 A	D5413	1	14					N				
-5 A	D5414	1	15					N				
-9 A	D5415	1	16					N				
-11 A	D5416	1	17					N				
-15 A	D5417	1	18					N				
-17 A	D5418	1	19					N				
-19 A	D5419	1	20					N				
Rinse	D5420	1	21					—	—	—		
	D5421	1	22					—	—	—		TTR 4/5/10

STANDARD	CODE
BFB	V3048
CAL. STD.	V341
LCS/MS MIX	V342
EXTRAS MIX	V3102

STANDARD	CODE
IS MIX	V3137
SS MIX	V3138

Circle Methods:
SW846 8260
EPA 624
EPA 524
SIM
OLM 04.2
OLM 03.1
OLC 02.1
OLC 03.2

0000072

TITLE: **ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

FIGURE 2

EXAMPLE OF GC/MS STANDARDS RECEIPT LOGBOOK PAGE

KATAHDIN ANALYTICAL SERVICES

STOCK STANDARDS RECEIVED

GCMS LABORATORY
REVIEWED BY/DATE:

AMP1518 1519 1520 1521 — 1-Chlorohexane (EPA-1208) Lot; CB-234, A Ultra Exp; 11/30/09 1000µg/mL — Rec'd 11/07/07

AMP1522 1523 1524 1525 — Restek Cat# 30434 MA VPH Standard 800–1500 ug/ml in Purge and Trap Methanol Lot# A040187 Exp: 11/12 Store: Freezer Restek Corporation - 110 Benner Circle - Bellefonte, PA 16823 — Rec'd 11/7/07 EKL

AMP1526 1527 — Restek Cat# 30465 RL Lot # A049252 Exp: 12/11 Store:Freezer — rec'd DMF 11/20/07

AMP1528 1529 — AccuStandard 125 Market St. - New Haven, CT 06513 - USA Tel: 203-786-5290 - www.accustandard.com FOR LABORATORY USE ONLY AS-E0285 Diethyl ether 5000 µg/mL in MeOH Lot: B4070099-1A Exp. Jan 19, 2010 1 mL STORAGE Ambient POISON — rec'd DMF 11/21/07

AMP1530 1531 — AccuStandard 125 Market St. - New Haven, CT 06513 - USA Tel: 203-786-5290 - www.accustandard.com FOR LABORATORY USE ONLY CLP-LC-IS-100X Laboratory Control Sample - Internal Standard Mix 2500 µg/mL in MeOH Lot: B2090027 Exp: Sep 6, 2012 1 mL 3 comps. STORAGE Ambient POISON

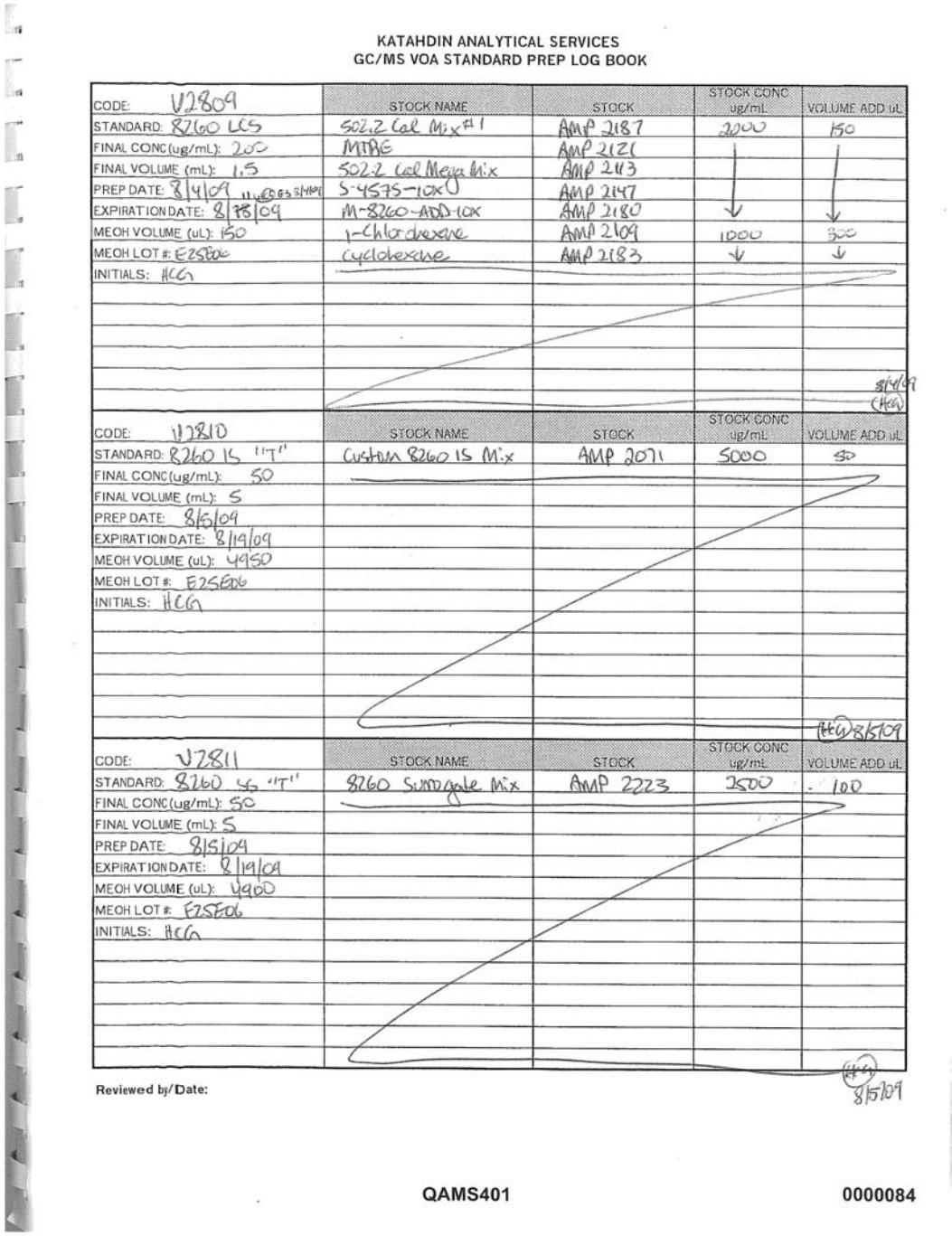
AMP1532 1533 — Restek Cat# 30624 RL Lot # A050631 Exp: 3/10 Store:Freezer — rec'd DMF 11/26/07

QAMS342 0000035

TITLE: **ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

FIGURE 3

EXAMPLE OF VOA STANDARDS PREPARATION LOGBOOK PAGE



KATAHDIN ANALYTICAL SERVICES
GC/MS VOA STANDARD PREP LOG BOOK

CODE: V2809	STOCK NAME	STOCK	STOCK CONC ug/mL	VOLUME ADD uL
STANDARD: 8260 LCS	502.2 Cal Mix #1	AMP 2187	2000	150
FINAL CONC (ug/mL): 20	MTBE	AMP 2121	↓	↓
FINAL VOLUME (mL): 1.5	502.2 Cal Mega Mix	AMP 2143		
PREP DATE: 8/4/09	5-4575-10X	AMP 2147		
EXPIRATION DATE: 8/18/09	M-8260-ADD-10X	AMP 2180	↓	↓
MEOH VOLUME (uL): 150	1-Chlorohexane	AMP 2109	1000	300
MEOH LOT #: E25E06	Cyclohexane	AMP 2183	↓	↓
INITIALS: HCG				
				8/4/09 (HCG)

CODE: V2810	STOCK NAME	STOCK	STOCK CONC ug/mL	VOLUME ADD uL
STANDARD: 8260 IS "T"	Custom 8260 IS Mix	AMP 2071	5000	50
FINAL CONC (ug/mL): 50				
FINAL VOLUME (mL): 5				
PREP DATE: 8/5/09				
EXPIRATION DATE: 8/19/09				
MEOH VOLUME (uL): 4950				
MEOH LOT #: E25E06				
INITIALS: HCG				
				(HCG) 8/5/09

CODE: V2811	STOCK NAME	STOCK	STOCK CONC ug/mL	VOLUME ADD uL
STANDARD: 8260 SS "T"	8260 Surrogate Mix	AMP 2223	2500	100
FINAL CONC (ug/mL): 50				
FINAL VOLUME (mL): 5				
PREP DATE: 8/5/09				
EXPIRATION DATE: 8/19/09				
MEOH VOLUME (uL): 4900				
MEOH LOT #: E25E06				
INITIALS: HCG				
				(HCG) 8/5/09

Reviewed by/Date:

QAMS401 0000084

FIGURE 4

TITLE: **ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

STANDARD INFORMATION

VOA Standards

Standard	Concentration	Manufacturer	Catalog Number
1,2,3 Trimethylbenzene	2000 ug/mL	Restek	58733
1,2,3 Trichlorobenzene	2000 ug/mL	Accustandard	M-502-47-10X
1,2,4 Trimethylbenzene	2000 ug/mL	Accustandard	M-502-54-10X
1,3,5 Trichlorobenzene	neat	Supelco	44-2235
1,3.5 Trimethylbenzene	2000 ug/mL	Accustandard	M502-55-10X
2-CEVE	2000 ug/mL	Accustandard	M-601C-10X
502.2 Cal Mix #1 (gases)	2000 ug/mL	Restek	30042
502.2 Cal2000 Mega Mix	2000 ug/mL	Restek	30431
504.1 Cal Mix	200 ug/mL	Accustandard	M-504.1-CSS
Acrolein & Acrylonitrile	5000 ug/mL	Accustandard	M-603-M-5X
Appendix IX Volatiles Mix	various	Accustandard	M-8240C-R3-10X
Bromochloromethane	2000 ug/mL	Accustandard	M-502-03-10X
California Oxygenates Mix #1	2000 - 10,000 ug/mL	Restek	30465
Carbon Disulfide	2000 ug/mL	Restek	30258
Chloroprene	2000 ug/mL	Accustandard	APPX9-048-R1
Custom GC Std	2000 ug/mL	Accustandard	S-11160
Custom VOC mix	various	Accustandard	S-7920-R1
Custom Volatile GC/MS Std	2000 ug/mL	Accustandard	S-3432B
Custom Volatiles GC/MS	2000 ug/mL	Accustandard	S-3432A
Diethey! Ether	5000 ug/mL	Accustandard	AS E0285
Freon 113	2000 ug/mL	Supelco	4-7944
Method 8260 Additions	2000 ug/mL	Accustandard	M-8260-ADD-10X
Method 8260B-Revision	2000 ug/mL	Accustandard	M-8240B-R-10X
MTBE	2000 ug/mL	Supelco	4-8483
Napthalene	2000 ug/mL	Accustandard	M-502-40-10X
THF	2000 ug/mL	Accustandard	S-4575-10X
Vinyl Acetate	2000 ug/mL	Restek	30216
Vinyl Acetate	2000 ug/mL	Accustandard	APPX9-211-20X
VOA Calibration Mix #1 (Ketones)	5000 ug/mL	Restek	30006
TCL Ketone Mix	5000 ug/mL	Accustandard	CLP-022-25X
VOC Liquid Mix	2000 ug/mL	Accustandard	M-502A-R2-10X
Volatile Organic Compounds (gases)	2000 ug/mL	Accustandard	M-502B-10X
IS/SS/Tune			
Custom 8260 IS	5000 ug/mL	Restek	54577
Custom 8260 SS	5000 ug/mL	Restek	54578
4-BFB	2000 ug/mL	Supelco	48083
VOA Tuning Compound (BFB)	5000 ug/mL	Restek	30003
1,2 Dichlorobenzene-D4	2000 ug/mL	Supelco	48952-U
Fluorobenzene	2000 ug/mL	Supelco	
VOA IS (CLP)	2500 ug/mL	Restek	30004
VOA SS (CLP)	2000 ug/mL	Supelco	48943
624 IS	1500 ug/mL	Restek	30023
4-BFB/Fluorobenzene/Pentafl. (EPA 624)	20000 ug/mL	Accustandard	M-624-SS-M
8260A SS	2500 ug/mL	Restek	30240
CLP Only			
04.1 CLP VOA Cal 2000	2000 ug/mL	Restek	30456
LCS-IS	2500 ug/mL	Accustandard	CLP-LCS-IS-100X
LCS-Volatiles	200 ug/mL	Accustandard	CLP-LCS-V
CLP Volatiles DMC Stock Solution	deutrated compds	Cambridge Isotope	ES 5038
3.2 OLC mix	1000 - 2000 ug/mL	Restek	30492

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-209
Revision History
Cover Page
Page 1

TITLE: ZERO HEADSPACE EXTRACTION (ZHE) OF VOLATILE SAMPLES FOR TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) METHOD 1311

Prepared By: GC/MS Group Date: 7/97

Approved By:

Group Supervisor: [signature] Date: 021301

Operations Manager: [signature] Date: 2/13/01

QA Officer: Deborah J. Nadeau Date: 2·12·01

General Manager: [signature] Date: 2/13/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 1311	Format changes, added pollution prevention. Changed wording around for section 7 and updated MS spiking.	DN	2·12·01	2·12·01
02 1311	added wording to section 8 minor changes to sections 8.2, 8.3 and table 1.	LAD	4/04	4/04
03 1311	grammatical and formatting correction	LAD	04/06	04/06
04 1311	Section 5.4 changed location of pH cal. LB. 4.3 removed acid washing procedure (req. for Metals only). 1.4 added waste stream information and proper disposal. Updated Tables & Figs. Added QC table, Analyte List (Table 3), Rotary Extractor Verification LB (Fig 2), Rotary Extractor (Fig 3) and ZHE Vessel (Fig 4). Added wording to Sect. 1e and 9. Added DOC info to Sect 8	LAD	03/07	03/07
05	Sect. 7.16 - corrected room temperature criteria. Table 2 - added method modifications for procedure of adding extraction fluid and the pressure the ZHE's are tumbled at. Updated logbook page.	LAD	11/08	11/08

TITLE: **ZERO HEADSPACE EXTRACTION (ZHE) OF VOLATILE SAMPLES FOR TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) METHOD 1311**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy _____ of document **SOP CA-209-05**, titled **Zero Headspace Extraction (ZHE) of Volatile Samples for Toxicity Characteristic Leaching Procedure (TCLP) Method 1311**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy _____ of document **SOP CA-209-05**, titled **Zero Headspace Extraction (ZHE) of Volatile Samples for Toxicity Characteristic Leaching Procedure (TCLP) Method 1311**.

Recipient: _____ Date: _____

TITLE: **ZERO HEADSPACE EXTRACTION (ZHE) OF VOLATILE SAMPLES FOR TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) METHOD 1311**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures used by Katahdin Analytical Services, Inc. technical personnel to extract solid matrix samples for volatile organics per EPA Method 1311, TCLP, using Zero Headspace apparatus.

1.1 Definitions

TCLP EXTRACTION BATCH: 20 or fewer samples, which are prepared together with the same method.

TCLP BLANK: An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. Baked organic-free sand is used as a blank matrix. The same extraction fluid used for the samples is used for the associated TCLP blank. The blank is taken through the appropriate steps of the process.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of all TCLP compounds are added to a sample matrix and a sample matrix duplicate after filtration of the TCLP extracts and prior to sample analysis. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision. The purpose of the matrix spike is to monitor the performance of the analytical methods used.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the Toxicity Characteristic Leaching Procedure by EPA Method 1311. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

It is the responsibility of all Katahdin technical personnel involved in the TCLP Method 1311 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Supervisor or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to indicate periodic review of the associated logbooks.

TITLE: **ZERO HEADSPACE EXTRACTION (ZHE) OF VOLATILE SAMPLES FOR TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) METHOD 1311**

1.3 Safety

When expressing the initial liquid from the waste to determine the percent solids, or when filtering the final TCLP extract from the ZHE after agitation, it is advisable to place the ZHE behind an explosion proof shield and to place the preweighed gas tight syringe on the liquid inlet/outlet valve without the plunger in the syringe. If the plunger is left in the syringe and the piston in the ZHE moves suddenly during pressurization, the plunger can become a dangerous projectile and/or the syringe could explode. Pressurize the ZHE slowly; if the pressure increases too much without the internal piston moving, carefully tap the outside of the ZHE to initiate movement. Do not exceed 20 psi if the piston does not move. In this event, vent the bottom flange and restart pressurization procedure. Too much pressure and a sudden release of the piston will force the liquid through the glass filter too fast, possibly rupturing the glass filter and/or blowing the syringe from the liquid inlet/outlet valve.

Always wear gloves, safety glasses and lab coat when handling the ZHE, sample or extract.

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures and the Katahdin Hazardous Waste Plan and follow appropriate procedures such as: wearing safety glasses and gloves when working with chemicals or near an instrument; not taking food or drink into the laboratory; each analyst should know the location of a respirator and be trained on how to use it properly and should know the location and use of all safety equipment.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Plan for further details on pollution prevention techniques.

Waste accumulated from the ZHE vessel is classified as organic soil waste stream “I,” which consists of leftover solids and used Borosilicate filters. The satellite for organic soil waste stream “I” is located inside the fume hood in the Volatile Organics Laboratory (room 111).

TITLE: **ZERO HEADSPACE EXTRACTION (ZHE) OF VOLATILE SAMPLES FOR TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) METHOD 1311**

Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Hazardous Waste Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

2.0 SUMMARY OF METHOD (taken from Method 1311)

For wastes containing greater than or equal to 0.5% solids, the liquid, if any, is separated from the solid phase and stored for later analysis. The particle size of the solid phase is reduced, if necessary. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. The extraction fluid employed is a function of the alkalinity of the solid phase of the waste. Following extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8 μm glass fiber filter.

For liquid wastes (i.e., those containing less than 0.5% dry solid material), the waste, after filtration through a 0.6 to 0.8 μm glass fiber filter, is defined as the TCLP extract. If compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

3.0 INTERFERENCES

Potential interferences that may be encountered during analysis are discussed in the individual analytical SOPs.

4.0 APPARATUS AND MATERIALS

4.1 The agitation apparatus must be capable of rotating the extraction vessel in an end-over-end fashion at 30±2 revolutions per minute (rpm) – see Figure 3. Each of the laboratory's rotary extractors is equipped with a device that displays the actual rotation rate in rpm. The rotation rate of each extractor is monitored before each use, and the measured rotation rates are recorded in a logbook maintained for that purpose (see Figure 2). If the measured rotation rate of an extractor is outside the range 30±2 rpm, it must be taken out of service until it can be repaired. (Associated Design and Manufacturing Co. or equivalent)

4.2 ZHE Vessels - The ZHE allows for initial liquid/solid separation, extraction and final extract filtration without opening the vessel – see Figure 4. The vessels have an internal volume of 500-600 mL and are equipped to accommodate a 90-110 mm filter. (Associated Design and Manufacturing Co. Model 3745-ZHE or equivalent).

4.3 Filters - Borosilicate glass fiber containing no binder materials having an effective pore size of 0.6 to 0.8 µm. (Environmental Express Cat. #FG7590MM, size - 90 mm, pore size - 0.7 µm, or equivalent). Prefilters must not be used. Glass fiber filters are fragile and should be handled with care.

4.4 pH Meters accurate to ±0.05 units at 25°C.

4.5 60 mL Gas-tight B-D Syringe - Collection device for initial liquid phase and the final extract of the waste when using the ZHE device.

4.6 ZHE Extraction Fluid Transfer Device - A 500 mL graduated cylinder which is capable of transferring the extraction fluid without changing the nature of it. (Associated Design and Manufacturing Co. Model #3775 or equivalent)

4.7 Laboratory Balance accurate to within ±0.1 grams (all weight measurements are to be within ±0.1 grams).

4.8 Beaker or Erlenmeyer flask, glass, 500 mL.

4.9 Magnetic stirrer.

4.10 Nitrogen tank complete with gauge as appropriate.

5.0 REAGENTS

Reagent grade chemicals shall be used in all tests. Other grades may be used only if it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. The purity of all chemicals must be known or evaluated before use to minimize any laboratory contamination.

5.1 Reagent water – Laboratory grade reagent water containing no interferent. Reagent water should be monitored periodically for impurities.

5.2 Sodium hydroxide (10N), NaOH, made from ACS reagent grade.

5.3 Glacial acetic acid, CH_3CH_2OOH, ACS reagent grade.

5.4 Extraction fluid 1 – Prepared and documented in Metals Laboratory. Please refer to the current revision of Katahdin Analytical Services SOP CA-510 for further information.

NOTE: The extraction fluid should be monitored frequently for impurities. The pH must be checked and documented prior to use to ensure that these fluids are made up accurately. Documentation of pH meter calibration prior to use is to be maintained in

the pH Meter Calibration Logbook (metals prep lab). If impurities are found in the extraction fluid or the pH of the fluid is not within 4.93 ± 0.05, the fluid shall be discarded and fresh extraction fluid prepared and documented.

5.5 Analytical standards shall be prepared according to the appropriate analytical method.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

All samples shall be collected in a soil jar using an appropriate sampling plan.

6.1 Sufficient sample must be collected to support the preliminary determinations and to provide an extract volume adequate for analytical and quality control purposes. The necessary sample size will depend on the solids content of the waste, but in no instance should less than 30 g of waste be provided to the laboratory.

6.2 Preservatives shall not be added to samples before extraction. Samples should be stored at 4°C (±2°C) and opened only immediately prior to TCLP extraction.

6.3 TCLP extracts should be prepared for analyses and analyzed as soon as possible following TCLP extraction. Sample holding times for Volatile TCLP extraction and analysis are as follows:

Date of sampling to TCLP extraction:	14 days
TCLP extraction to analysis:	14 days

7.0 PROCEDURES

SAMPLE PREPARATION

7.1 Adjust the piston within the ZHE to a height that will minimize the distance it will have to move once the ZHE is charged with sample. It may be necessary to moisten the O-rings with extraction fluid to adjust the piston.

7.2 Weigh out a 10 g subsample of the waste and record the weight in the Volatile TCLP Extraction Logbook (Figure 1).

7.3 If the waste will obviously yield no liquid when subjected to pressure filtration, i.e., appears to be 100% solids, proceed to 7.12.

7.4 If the sample appears to contain low total solids (high degree of moisture but greater than .5% solids), approximate the amount of waste necessary so that after the liquid has been expressed there will be approximately 10 g of solid waste in the vessel and continue with step 7.7. The vessel can only be charged once.

TITLE: **ZERO HEADSPACE EXTRACTION (ZHE) OF VOLATILE SAMPLES FOR TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) METHOD 1311**

$$\text{Weight of waste to charge ZHE} = \frac{10}{\%\ \text{solids}} \times 100$$

Generally, the TCLP Metals group will determine the percent solid of the sample and those results can be used in lieu of proceeding with the determination in step 7.7.

7.5 If the sample is less than .5% solids, it will be run as an aqueous sample. If the sample appears to be less than 0.5 % dry solids, this filtrate (or sample) is defined as the TCLP extract and is analyzed directly (See section 7.18).

7.6 If the sample appears to be an oil, extract as a medium level soil.

ZHE WITH PRELIMINARY DETERMINATION OF PERCENT SOLIDS

7.7 Quantitatively transfer the entire sample (both liquid and solid phases) quickly to the ZHE. Place the filter and support screens onto the top flange of the device and tighten. If it appears that more than 1% of the original sample weight has adhered to the container, determine the weight of this residue and subtract it from the sample weight.

7.8 When expressing the initial liquid from the waste to determine the percent solids, or when filtering the final TCLP extract from the ZHE after agitation, it is advisable to place the ZHE behind an explosion proof shield and to place the preweighed gas tight syringe on the liquid inlet/outlet valve without the plunger in the syringe. If the plunger is left in the syringe and the piston in the ZHE moves suddenly during pressurization, the plunger can become a dangerous projectile and/or the syringe could explode. Pressurize the ZHE slowly; if the pressure increases too much without the internal piston moving, carefully tap the outside of the ZHE to initiate movement. Do not exceed 20 psi if the piston does not move. In this event, vent the bottom flange and restart pressurization procedure. Too much pressure and a sudden release of the piston will force the liquid through the glass filter too fast, possibly rupturing the glass filter and/or blowing the syringe from the liquid inlet/outlet valve.

7.9 Attach a preweighed collection syringe to the liquid inlet/outlet valve (top flange) and open the valve. Attach the gas line to the gas inlet/outlet valve and pressurize to 1-10 psi slowly. Carefully increase the pressure to 50 psi at 10 psi increments (monitor collection syringe to prevent excessive pressure buildup which could detach or break the syringe). At each 10 psi increment, wait 2 minutes for additional liquid flow. Stop filtration when liquid flow has ceased within a 2 minute period at 50 psi.

CAUTION: Too much pressure at once can degrade the glass fiber filter and may cause premature plugging.

7.10 Reweigh the collection syringe to determine the percent solid. The material in the ZHE is defined as the solid phase of the waste and the filtrate is defined as the liquid phase.

$$\% \text{ Solids} = \frac{\text{Weight of initial sample - liquid}}{\text{Total weight of waste}} \times 100$$

The liquid phase may be analyzed immediately or stored at 4°C until time of analysis.

7.11 The particle size of a sample must be smaller than 1 cm in diameter prior to extraction. If particle size reduction is necessary, it can be accomplished by crushing, grinding or cutting particles that do not meet the size criteria. However, the sample and reduction equipment must be refrigerated to 4°C before size reduction.

Sieving the waste may cause volatiles to be lost and thus is not recommended. Proceed with step 7.15.

ZHE FOR WASTES WITH 100% PERCENT SOLIDS

7.12 The particle size of a sample must be smaller than 1 cm in diameter prior to extraction. If particle size reduction is necessary, it can be accomplished by crushing, grinding or cutting particles that do not meet the size criteria. However, the sample and reduction equipment must be refrigerated to 4°C before size reduction.

Sieving the waste may cause volatiles to be lost and thus is not recommended.

7.13 Quantitatively transfer the entire sample quickly to the ZHE. Place the filter and support screens onto the top flange of the device and tighten. If it appears that more than 1% of the original sample weight has adhered to the container, determine the weight of this residue and subtract it from the sample weight.

7.14 Determine the amount of extraction fluid #1 to add to the ZHE. If the waste appears to be less than 0.5 % liquid, or basically dry, use 10 g waste and 200 mL extraction fluid #1.

$$\text{amt. of extraction fluid} = \frac{20\ (\%\ \text{solids})\ (\text{weight of waste filtered})}{100}$$

SAMPLE EXTRACTION

7.15 If the ZHE has been pressurized (determination of percent solids), release gas pressure on the ZHE piston. Add the appropriate amount of extraction fluid #1 using the 500 mL graduated cylinder. If the extraction fluid was prepared on the same day as sample extraction, ensure that fluid prep information has been recorded on the bench sheet in the Volatile TCLP Extraction Logbook. Otherwise, reference the date of fluid preparation on the bench sheet. Check the ZHE to ensure that there are no leaks. Pressurize the ZHE with 5-10 psi and check again for leaks. When the

pressure is at 10 psi and no leaks appear, slowly open the liquid inlet/outlet valve to bleed out any headspace that may have been introduced during the addition of extraction fluid. Stop the bleeding at the first appearance of liquid from the valve since the appearance of liquid is an indicator of no headspace.

7.16 Place the ZHE in the rotary agitation apparatus and rotate at 30±2 rpm for 18±2 hours. Record the TCLP extraction start and end time in the Volatile TCLP Extraction log. Room temperature must be maintained and documented to be at 23 ± 2°C during agitation.

7.17 After the 18±2 hour agitation period, check the pressure gauge on the ZHE to ensure there were no leaks over that time period. If the pressure within the device has been maintained, the sample in the ZHE vessel is separated into its component liquid and solid phases, as discussed in Steps 7.8 - 7.9. The liquid is carefully dispensed from the collection syringe into a VOA vial at a rate that precludes effervescence.

If the original waste contained no initial liquid phase, the filtered liquid material obtained is defined as the TCLP extract. If the waste contained an initial liquid phase, the filtered liquid material obtained and the initial liquid phase are collectively defined as the TCLP extract.

If the individual phases are analyzed separately determine the volume of the individual phase (to 0.5%), conduct the appropriate analyses, and combine the results mathematically by using a simple volume weighted average, as follows:

$$\text{Final Analyte Concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

Where: V_1 = The volume of the first phases (L).
C_1 = The concentration of the analyte of concern in the first phase (mg/L).
V_2 = The volume of the second phase (L).
C_2 = The concentration of the analyte of concern in the second phase (mg/L).

If the individual liquid phases are compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the TCLP extract and these are analyzed together.

7.18 Refer to Katahdin SOP CA-202, Analysis of VOAs By SW-846 Method 8260, current revision, for detailed procedure for GC/MS calibration and analysis.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

TITLE: **ZERO HEADSPACE EXTRACTION (ZHE) OF VOLATILE SAMPLES FOR TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) METHOD 1311**

Refer to the current revision of Katahdin SOP CA-202, Analysis of VOAs by Method 8260, for applicable quality control criteria. The following QC samples are prepared with each TCLP extraction batch:

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 Blanks: 1 blank is run per set per day. 1 ZHE vessel is loaded with 10 grams of baked sand, 200 mL extraction fluid #1, and analyzed as if it were a regular sample.

8.2 One Matrix Spike and Matrix Spike Duplicate extraction is performed for every 20 extractions. Matrix spike solution is added after filtration of the TCLP extract. All TCLP compounds are spiked.

Matrix spikes are added at a concentration equivalent to the corresponding regulatory limit, but not less than 5 times the method detection limit.

Matrix spike recoveries are calculated by the following formula:

$$\%R\ (\%\ \text{Recovery}) = 100\ (X_s - X_u)/K$$

Where: X_s = measured value for the spiked sample,
X_u = measured value for the unspiked sample, and
K = known value of the spike in the sample.

Measured sample values are reported without correction for analytical bias (based on the matrix spike recovery).

8.2.1 Preparation of Matrix Spike: The matrix spike is prepared with the method 8260 LCS mix. Refer to the 8260 SOP CA-202, current revision for further details.

8.2.2 Each new analyst must demonstrate her/his ability to perform the method acceptably by while being witnessed by an analyst who is experience in performing the method. To successfully demonstrate the method, the analyst must perform the method in conformance with all the requirements of the SOP, referring to the SOP for guidance as necessary. In addition, each analyst must demonstrate the ability to produce TCLP Extraction Blanks that are free

of contamination. This demonstration will require the analyst to collect and file the analytical results from four Extraction Blanks that he/she has generated

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Department Manager and with the QAO.

Refer to the current revisions of USEPA Method 1311 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste (SW-846), Third Edition, Final Update III, Method 1311, US EPA, 12/96 or current revision

LIST OF TABLES AND FIGURES

TITLE: **ZERO HEADSPACE EXTRACTION (ZHE) OF VOLATILE SAMPLES FOR TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) METHOD 1311**

TABLE 1

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Toxicity Characteristic Leaching Procedure (TCLP)/ EPA 1311	Method Blanks	One per 20 samples extracted using a particular batch of extraction fluid.	Refer to individual analytical method.	Prepare fresh extraction fluid and repeat TCLP extraction of all associated samples.
		One per 20 samples extracted in a particular extraction vessel.	Refer to individual analytical methods.	Remove extraction vessel from service.
	Matrix Spike	One per 20 TCLP extractions performed (required). One per waste type (suggested, left to discretion of client).	Refer to individual analytical method.	Refer to individual analytical method.
	Demonstration of analyst proficiency; accuracy and precision	One time demonstration by each analyst performing the method	New analyst's performance of the method is witnessed by an experienced analyst. New analyst must produce method blanks that meet all method and laboratory acceptance criteria.	Repeat analysis until able to demonstrate acceptable performance of the method to witnessing analyst and by producing acceptable method blanks; document successful performance in personal training file.

TITLE: **ZERO HEADSPACE EXTRACTION (ZHE) OF VOLATILE SAMPLES FOR TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) METHOD 1311**

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-209-05	METHOD 1311, current revision
Apparatus/Materials		
Reagents		
Sample preservation/ handling	Leachate is drawn into a 60 mL gas tight syringe and is dispensed into VOA vials at a rate that precludes effervesence.	Method recommends the use of TEDLAR bags and or 600 mL gas tight syringes.
Procedures	Preliminary TCLP evaluations done on 10 g aliquot of waste; this subsample is also used for TCLP extraction. Extraction fluid is added to the ZHE prior to addition of sample ZHEs are tumbled at 40 ± 2 psi	Preliminary TCLP evaluations done on minimum 100 g aliquot of waste, which may not actually undergo TCLP extraction. Sample size for TCLP extraction is based on 25 g of solid in the waste subsample. Extraction fluid is added through the inlet valve. ZHEs are tumbled at 5 - 10 psi
QC - Method Blanks	Frequency of one method blank per 20 extractions or each batch.	Frequency of one method blank per 20 extractions performed in a particular extraction vessel.
QC – Spikes	One Matrix Spike and Matrix Spike Duplicate extraction is performed for every 20 extractions.	A matrix spike shall be performed for each waste type (e.g., wastewater treatment sludge, contaminated soil, etc.) unless the result exceeds the regulatory level and the data are being used solely to demonstrate that the waste property exceeds the regulatory level. A minimum of one matrix spike must be analyzed for each analytical batch. As a minimum, follow the matrix spike addition guidance provided in each analytical method.
QC – LCS		
QC – Accuracy/Precision		
QC – MDL		

TITLE: **ZERO HEADSPACE EXTRACTION (ZHE) OF VOLATILE SAMPLES FOR TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) METHOD 1311**

TABLE 3

TOXICITY CHARACTERISTIC CONSTITUENTS AND REGULATORY LEVELS

Constituent	Regulatory Level (mg/L)
Benzene	0.5
Carbon tetrachloride	0.5
Chlorobenzene	100.0
Chloroform	6.0
1,2-Dichloroethane	0.5
1,1-Dichloroethene	0.7
Methyl ethyl ketone	200.0
Tetrachloroethene	0.7
Trichloroethene	0.5
Vinyl Chloride	0.2

TITLE: **ZERO HEADSPACE EXTRACTION (ZHE) OF VOLATILE SAMPLES FOR TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) METHOD 1311**

FIGURE 1

EXAMPLE OF VOLATILE TCLP EXTRACTION LOGBOOK PAGE

KATAHDIN ANALYTICAL SERVICES, INC.
VOLATILE TCLP EXTRACTION BENCH SHEET FOR 586578 - 6A

Dry Weight Determination 100% ✓ <0.5%_____ %Wet Solids_____

_____Refer to non-volatile TCLP extraction sheet, page #_____ for _____-_____

Sample description:_____Homogeneous_____ Non-homogeneous_____

1)	Weight Container 1 + Residue	_____g	7)	Weight of Filter	_____g
2)	Weight Container 1 + Waste	_____g	8)	Wt of Filter and Wet Solid	_____g
3)	Weight of Waste ((2) - (1))	_____g	9)	Wt of Wet Solid Phase ((8) - (7))	_____g
4)	Weight of Container 2	_____g	10)	%Wet Solids (100*(9)/(3))	_____
5)	Weight of Container 2 and Filtrate	_____g	11)	Wt of Filter + Solid Phase Dry	_____g
6)	Wt(g)/Vol(mL) of Filtrate ((5) - (4))	___1___	12)	Wt of Solid Phase Dry ((11) - (7))	_____g
			13)	%Dry Solids (100*(12)/(3))	_____

Analyst TTC Date 11/19/08

Extraction Fluid Preparation and pH Check Fluid #1 Prepared Today_____ (Complete Prep Info Below)
Date prepared (if previously prepared) 11/11/08 Refer to Prep Info on Page Meghs Prep

_____mL glacial acetic acid, manuf./lot #_____, added to 500 mL reagent water; _____mL ____N NaOH, manuf./lot #_____ added, then solution diluted to 1 L with reagent water.

Analyst TTC Date 11/19/08 pH of Fluid Today 4.93 Extraction Fluid Batch # 878

Extract Use Filter Paper Lot #: 53.5040/

___<0.5% Solids (Filter adequate sample volume) _____mL Filtered.
___✓100% Solids
10 g of Waste added to 200 mL of fluid (fluid volume = 20 times wt of solid sample)
___ Free Liquid present (See Equation A for amount of waste to filter)

(A) X = Desired weight of solid phase on filter * 100/ (%Wet Solid)

1)	Weight Container 1 + Waste		_____g
2)	Weight Container 1 + Residue		_____g
3)	Weight of Waste Charged to ZHE ((1) - (2), or weighed directly into vessel)		_____g
4)	Weight of Pre-extraction Collection Device		_____g
5)	Weight of Device and Filtrate		_____g
6)	Weight ((5) - (4))/Volume (mL) of Filtrate	6a_____/_____6b	
7)	_____g of Solid Phase was added to 8)_____mL of Fluid #1. ((3) - (6a)) (20 * (7a))		

Pressure before Tumbling 37 Pressure after Tumbling: 39
Time Started Tumbling 15:52 Time Stopped 10:40 Hours Extracted 18.48
Room Temp @ Start 21 Room Temp @ End _____

Was pre-extracted filtrate recombined with extract _____YES _____NO
If "NO" enter volume of filtrate (6) _____
Amount of Fluid (8) _____

Analyst TTC Date 11/20/08 Rev 6/08

QAMS383 000009

FIGURE 2

TITLE: **ZERO HEADSPACE EXTRACTION (ZHE) OF VOLATILE SAMPLES FOR TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) METHOD 1311**

EXAMPLE PAGE FROM ROTARY EXTRACTOR RPM VERIFICATION LOGBOOK

KATAHDIN ANALYTICAL SERVICES, INC.

ROTARY EXTRACTOR RPM VERIFICATION LOGBOOK

EXTRACTOR #1: TOP SHELF SERIAL NUMBER: NONE
EXTRACTOR #2: MIDDLE SHELF SERIAL NUMBER: 1173
EXTRACTOR #3: BOTTOM SHELF SERIAL NUMBER: 1169

Please record the number of RPMs for each extractor each time they are used.

Date	Initials	Extractor #1	Extractor #2	Extractor #3	Comments
10/26/06	ars	out of service	30	30	Replaced fuse in Ext. #2
10/	ALL	out of service	29	Noise	
11/1/06	DMF	↓	30	↓	
11/30/06	DMF	↓	not in use	30	
12/27/06	DMF	↓	↓	30	
01/09/07	DMF	↓	↓	30	
01/11/07	DMF	↓	↓	30	
01/22/07	DMF	↓	↓	29	
01/25/07	DMF	↓	↓	30	
01/29/07	DMF	↓	↓	30	
01/31/07	DMF	↓	↓	30	
02/06/07	DMF	↓	↓	30	
02/12/07	DMF	↓	↓	30	

Acceptance Range is 28-32 RPMS.
Meters Should Be Verified Against A Wrist Watch Annually And Recorded In The Comments Section.

QAQC276 **0000003**

FIGURE 3

ROTARY AGITATION APPARATUS

TITLE: **ZERO HEADSPACE EXTRACTION (ZHE) OF VOLATILE SAMPLES FOR TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) METHOD 1311**

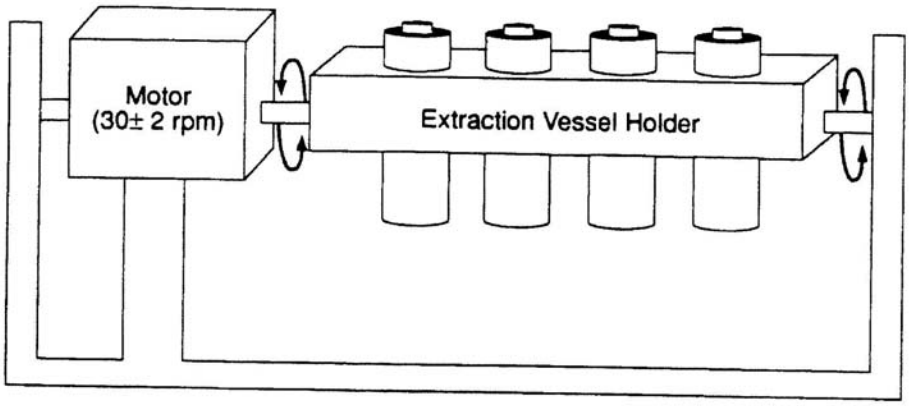


Figure 1. Rotary Agitation Apparatus

FIGURE 4

TITLE: **ZERO HEADSPACE EXTRACTION (ZHE) OF VOLATILE SAMPLES FOR TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) METHOD 1311**

ZHE VESSEL

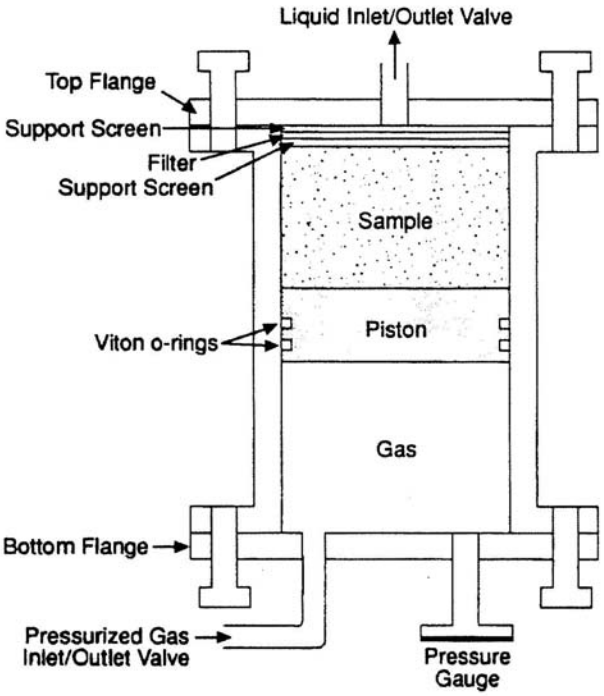


Figure 2. Zero-Headspace Extractor (ZHE)

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

Prepared By: GC/MS Department Date: 6/98

Approved By:

Group Supervisor: J. Halay Date: 020101

Operations Manager: John C. Benton Date: 1/31/01

QA Officer: Deborah J. Nadeau Date: 1.31.01

General Manager: Duncan F. Hughan Date: 2/01/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 8270C Mod.	Format changes, added pollution prevention, added instrument and other calibration options. Other minor changes to sections 7, 8 & QA Table.	DH	1.31.01	1.31.01
02 8270C	Many changes in formatting. Some additions to sections & Table 1 to comply with Navy.	DH	09.30.04	09.30.04
03 8270C	Sect. 7.2: Removed "K" Instrument & added "R" instrument. Added Pentafluorophenol surr. to Tables 3, 5 and Sect. 8.2. Removed all References to TIC's.	LAD	04/06	04/06
04 8270C	Sect. 8.2 - changed 5 to 4 and removed pentachlorophenol. Table 3 and 5 - removed pentachlorophenol. Changed linear regression correlation coefficient criteria. Added MI SOP reference. Added LCS exceedance criteria. Added ICV requirement and criteria. Added RT window procedure.	LAD	06/07	06/07
05 8270C	Added "G" instrument, Removed "X" instrument Edited Section 7.5.1 - initial cal. table	LAD	02/08	02/08

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Section 5.3.2.3- Added calibration Mix B. Section 7.5.1- Edited to address diffent SIM compounds may need to be calibrated at different levels depending on the compound and project requirements.	LAD	04/09	04/09
07	Changes made for compliance with DoD QSM version 4.1	LAD	08/09	08/09
08	Updated Standard prep. Added compounds to Table 3 and 5. Updated references. Added DoD QSM QC requirements Table.	LAD	04/10	04/10

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document SOP CA-213-08, titled "**ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY METHOD 8270 – Modified for Selected Ion Monitoring (SIM)**".

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document SOP CA-213-08, titled "**ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY METHOD 8270 – Modified for Selected Ion Monitoring (SIM)**".

Recipient: _____ Date: _____

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures utilized by Katahdin Analytical Services, Inc. laboratory personnel to prepare and analyze water and soil sample extracts for semivolatile organics by EPA SW-846 Method 8270, current revision, modified for selected ion monitoring.

In order to maintain consistency in data quality, this SOP consolidates all aspects of the analyses in one working document, to be revised as necessary.

1.1 Definitions

ANALYTICAL BATCH: 20 or fewer samples which are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods.

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

CALIBRATION CHECK: Verification of the ratio of instrument response to analyte amount, a calibration check is done by analyzing for analyte standards in an appropriate solvent. Calibration check solutions are made from a stock solution that is different from the stock used to prepare standards.

CALIBRATION STANDARD (WORKING STANDARD): A solution prepared from the stock standard solution that is used to calibrate the instrument response with respect to analyte concentration.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

STANDARD CURVE (CALIBRATION CURVE): A curve that plots concentration of known analyte standard versus the instrument response to the analyte.

STOCK STANDARD SOLUTION: A concentrated solution containing a single certified standard that is a method analyte, or a concentrated solution of a single analyte prepared in the laboratory with an assay reference compound. Stock standard solutions are used to prepare calibration standards.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

TARGET: A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

TARGET DB: An oracle database used to store and organize all Target data files.

QUICKFORMS: A laboratory reporting software for Target and Target DB. The QuickForms report module for Target is preconfigured with generalized forms and US EPA CLP report forms and disk deliverables, which can be customized.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of semivolatile organic compounds by EPA Method 8270. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, “Personnel Training & Documentation of Capability,” current revision.

It is the responsibility of all Katahdin technical personnel involved in analysis of semivolatiles by Method 8270 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with the Katahdin Analytical Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves, and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

After analysis, autosampler vials containing sample extracts in methylene chloride are returned to the SVOA hood, and the contents transferred to a labeled waste container. The contents of this container are disposed of in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

2.0 SUMMARY OF METHOD

The process involves the extraction of semivolatiles from a sample using an appropriate solvent followed by clean up steps (where applicable) and concentration of the extract (refer to Katahdin SOP CA-502, “Preparation of Aqueous Samples for Extractable Semivolatile Analyses”, SOP CA-512, “Preparation of Sediment/Soil Samples by Sonication Using Method 3550 for Subsequent Extractable Semi-Volatiles Analysis” and SOP CA-526, “Preparation of Sediment/Soil Samples by Soxhlet Extraction Using Method 3540 for Subsequent Extractable Semivolatile Analysis”). An aliquot of the final extract is injected into the gas chromatograph for compound separation by capillary column, followed by the electron impact mass spectrometer for identification and quantitation.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

3.0 INTERFERENCES

Interfering contamination may occur when a sample containing low concentrations of SVOCs is analyzed immediately after a sample containing high concentrations of SVOCs. Any samples that have suspected carryover must be reanalyzed.

4.0 APPARATUS AND MATERIALS

4.1 GC: Hewlett Packard 5890 and/or 6890

4.2 Mass Spectrometers (MS): HP5973, HP5972 and/or HP5970

4.3 Helium: Carrier gas for routine applications. All carrier gas lines must be onstructed from stainless steel or copper tubing; non-polytetrafluoroethylene (non-PTFE) thread sealant or flow controllers with rubber component are not to be used.

4.4 Autosamplers: HP 7673As

4.5 Hamilton syringes: 2.00 uL to 10 mL

4.6 Volumetric glassware: Grade A or equivalent

4.7 Columns: DB-5MS 30m, 0.25mm I.D., 25um film thickness, columns (J&W Scientific) or equivalent.

4.8 Acquisition System: The acquisition system must be interfaced to the MS and allow continuous acquisition of data throughout the duration of the chromatographic program. It must permit, at a minimum, the output of time vs. intensity (peak height or peak area). Hewlett Packard Chemstation or equivalent.

4.9 Data System: The Target software is used for processing data and generating forms.

5.0 REAGENTS

5.1 J.T. Baker Ultra Resi-Analyzed methylene chloride (or equivalent)

5.2 Purge and trap grade methanol

5.3 Standards: Stock standards and working standards are received and recorded in accordance with SOP CA-106 “Standard Preparation and Documentation”.

5.3.1 The expiration date for all standards is one year from date of opening the ampule. If the manufacturer’s expiration date is before this one year date,

the manufacturer's expiration must be followed. New standards must be opened if degradation is observed.

5.3.2 Secondary dilution standards

5.3.2.1 The standards are prepared on an as needed basis (or every 6 months) and stored in screw-cap amber bottles with Teflon liners in the BNA standards freezer between uses. Standards prepared from various stock solutions must always use the first expiration date of any of the solutions used for preparation.

5.3.2.2 Calibration Mix A – Prepare standards in methylene chloride containing the compounds listed in Table 3. The final concentration of each compound is 20 ug/mL.

5.3.2.3 Calibration Mix B - Some compounds must be calibrated at higher concentrations. For these compounds a secondary standard is prepared which will "boost" the concentration of these compounds in the initial calibration. The concentration of this standard is determined on a project to project basis.

5.3.2.4 Internal Standard Solution – Prepare standard in methylene chloride containing 1,4-dichlorobenzene-d4, naphthalene-d8, acenaphthene-d10, phenanthrene-d10, chrysene-d12, and perylene-d12 at a final concentration of 80 ug/mL.

5.3.2.5 DFTPP Solution – Prepare standard in methylene chloride containing DFTPP at a final concentration of 25 ug/mL.

5.3.2.6 Independent Calibration Verification (ICV) Standard – From a source independent of the calibration standards, prepare a standard in methylene chloride containing the compounds listed in Table 3. The final concentration of each compound is 2 ug/mL.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

All semivolatile sample extracts must be analyzed within forty days following the date of extraction.

7.0 PROCEDURES

7.1 NAMING AND CODING CONVENTIONS FOR ANALYTICAL STANDARDS – Used in accordance with SOP CA-106 "Standard Preparation and Documentation".

7.2 COMPUTER (DATA SYSTEM) CONVENTIONS -

Conventions for all instruments are as follows:

Sub-Directory for data acquisition and storage: C:\HPCHEM\1\DATA
Tune file: DFTPP.U

Method files: LSPSIMXX.M (all samples and standards)
Where:
XX = the calibration number in chronological order
L = instrument ID (R, U, or G)
DFTPP390.M (DFTPP tuning acquisition)

NOTE: All acquisition parameters must be identical for LSPSIMXX.M and DFTPP2. M.

Data Files: L_ _ _ _.D, where _ _ _ _ is a number in chronological order from 0001 to 9999 and L is the instrument ID (R, U, or G). This file also contains the Quantitation output file.

Data Files for DFTPP: LD_ _ _.D, where _ _ _ is a number in chronological order from 001 to 999 and L is the instrument ID (R, U, or G).

7.3 INSTRUMENT SPECIFIC PROCEDURES

It is the policy of the GC/MS group that all data be acquired in the batch mode. The following items must be checked prior to data acquisition in the batch mode:

- Ensure that the proper sequence and tune files are being used.
- Check the autosampler syringe (Is it clean? Does the plunger move freely? etc.), its alignment and make sure the solvent rinse vial is full. Ensure that the knurled nut holding the top of the syringe plunger is tight.
- Look at the batch to be analyzed and check the following:

  -Make sure that the data files are in numerical order with no duplication and that the method file is the same as that used for ICAL or Continuing Calibration analysis.

  -Bottle numbers match with the numbers on the autosampler tray.

After the batch has been deemed free of errors, start the batch by using the “Position and run” command under the SEQUENCE menu in MSTop.

7.4 INSTRUMENT TUNING - Prior to the analysis of any calibration standards, blanks or samples, the GC/MS system must be shown to meet the mass spectral key ion and ion abundance criteria for decafluorotriphenylphosphine (DFTPP) tabulated below. Pentachlorophenol, benzidine and DDT are also present in this standard.

DFTPP Key Ions and Ion Abundance Criteria	
Mass	**Criteria**
51	30.0-80.0 percent of mass 198
68	less than 2.0 percent of mass 69
69	present
70	less than 2.0 percent of mass 69
127	40.0 – 60.0 percent of mass 198
197	less than 1.0 percent of mass 198
198	base peak, 100 percent of mass 198
199	5.0-9.0 percent of mass 198
275	10.0-30.0 percent of mass 198
365	greater than 1.00 percent of mass 198
441	present, but less than mass 443
442	greater than 40.0 percent of mass 198
443	17.0-23.0 percent of mass 442

All ion abundances must be normalized to m/z 198, the nominal base peak.

The following are the GC/MS operating conditions for injection of DFTPP.

GC/MS Operating Conditions - DFTPP	
Initial column temperature hold	140°C for 3 minutes
Column temperature program	140-275°C at 15 degrees/minute
Final column temperature hold	275°C
Injection port temperature	280°C
Transfer line/source temperature	285°C
Injector - splitless, valve time	0.18 minutes
EPC	inlet B
Constant flow	ON
Constant flow pressure	10psi
Constant flow temperature	30°C
Vacuum comp.	ON
Run time	10-12 minutes
Scan start time	5.0 minutes
Sample volume	2.0 uL of 25 ng/uL DFTPP solution
Carrier gas	helium at @ 1.0 mL/minute
Mass range	35 to 500 amu
Number of A/D samples	4
GC Peak threshold	500 counts
Threshold	10 counts

Set up the run on the Enviroquant system using "Edit Sample Log Table". For a more detailed explanation of the Enviroquant software, consult the appropriate manual, Organic Department Manager, or senior chemist within the GC/MS group.

The DFTPP solution must be analyzed once at the beginning of each twelve hour period during which standards and/or samples are analyzed. The 12 hour time period for GC/MS system begins at the moment of injection of the DFTPP analysis. The time period ends after twelve hours has elapsed according to the system clock. The last injection must be accomplished prior to the expiration of 12 hours; conceivably, the run-time of an injection could end after the twelve hours.

When the DFTPP has concluded, the run must be evaluated to determine if sample analysis can proceed. The chromatography and the ion ratios must be examined. The DFTPP run is processed using the current algorithms in the Target software.

If the results indicate the system does not meet acceptance criteria, the GC/MS must be manually tuned. Once the manual tune procedure is completed, DFTPP must be re-injected and reevaluated. If the instrument still does not meet criteria, notify your Department Manager. Under no circumstances should calibration proceed if the instrument DFTPP is not in criteria.

7.5 INSTRUMENT CALIBRATION

7.5.1 Initial Calibration for Method 8270-SIM

Prior to the analysis of samples and required method blanks, and after the instrument DFTPP tuning criteria have been met, the GC/MS system must be calibrated at six different concentrations, typically, 0.20, 0.50, 1.0, 2.0, 5.0 and 8.0 ng/uL. This is done to determine instrument sensitivity and the linearity of GC/MS response for the semivolatile target and surrogate compounds.

Some SIM compounds may need to be calibrated at higher concentrations. A second standard is prepared containing these compounds. The two standards are combined as in the example below. A 100 uL aliquot of each of the standards above is spiked with 1 uL of internal standards and analyzed.

Example –
For a calibration at the following levels:
Calibration mix A would be prepared containing ALL analytes at 20 ng/ul
Calibration Mix B would be prepared containing phenols and phthalates at 20 ng/ul.

Final PAH conc. (ng/uL)	Final Conc. Phenols and phthalates (ng/ul)	Cal-Mix A Added (uL)	Cal-Mix B Added (uL)	$MeCl_2$ Added (uL)	Final Volume (uL)
0.20	1.0	10	40	950	1000
0.50	2.0	25	75	900	1000
1.0	3.0	50	100	850	1000
2.0	4.0	100	100	800	1000
5.0	5.0	250	0	750	1000
8.0	8.0	400	0	600	1000

Note: Calibration Mix B only is used to boost the phenols and phthalates concentrations in Cal. levels 1 through 4.

The GC/MS operating conditions for the calibration standards injections are the same as for the DFTPP with the following exceptions:

GC/MS Operating Conditions – Calibration and Samples	
Column temperature program	40°C for 3 min. to 300°C at 10°/min.
Final column temperature hold	300°C
Run time	35 minutes (time may vary dependent upon column length)
Scan start time	2.0-6.0 minutes (time may vary dependent upon column length)
Sample volume	1 uL

The conditions are set up in the method file LSPSIMXX.M

After analysis of the five calibration points, they must be quantitated and evaluated for adherence to QC criteria. Minimum requirements of ID files are the use of specific quantitation ions and quantitating a specific set of targets and surrogates with a set internal standard. Of particular importance when performing SIM analysis are the ion ratios. These requirements are found in Tables 3 and 5.

7.5.2 Initial Calibration Criteria

Relative response factors (RRFs) must be calculated and evaluated for each target compound and surrogate. The RRF is defined as follows:

$$RRF = \frac{Ax}{A_{IS}} \times \frac{C_{IS}}{Cx}$$

where: A_x = area of the primary ion for the target compound
A_{IS} = area of the primary ion for the corresponding istd
C_{IS} = concentration of the istd (ng/uL)
C_x = concentration of the target compound

After the calibration points have been quantitated, update the calibration curve points using the Target data processing software to generate the RRF's and %RSD's for all analytes. If information is needed concerning the use of these programs, consult the Organic Department Manager or a senior chemist within the group.

Response factor criteria have been established for the calibration of the semivolatile target and surrogate compounds. These criteria must be met in order for the calibration curve to be considered valid. The percent RSD for each calibration check compound (CCC) must be less than or equal to 30 percent. There are three CCC's: Acenaphthene, Fluoranthene, and Benzo(a)pyrene. There are no criteria for the SPCC compounds. This is also applicable to clients that request DOD criteria.

7.5.2.1 Linearity of Target Analytes (This is also applicable to clients that request DOD criteria.)

If the RSD of any target analyte is 15% or less, then the response factor is presumed to be constant over the calibration range, and the average response factor may be used for quantitation.

If the RSD of any target analyte exceeds 15%, then a calibration option outlined in section 7.0 of method 8000 will need to be employed.
Option 1 (Section 7.5.2 of method 8000 - Rev. 2, 12/96), is a linear regression of instrument response versus the standard concentration.

The correlation coefficient (r) for each target analyte and surrogate must be greater than or equal to 0.995. Target software calculates the correlation coefficient squared (r^2). This must be equal to or greater than 0.990.

Option 2 (Section 7.5.3 of method 8000 - Rev. 2, 12/96), is a non-linear calibration model not to exceed a third order polynomial. The lab would use a quadratic model or second order polynomial. The use of a quadratic model requires six calibration points. In order for the quadratic model to be acceptable, the coefficient of determination must be greater than or equal to 0.990.
If time remains in the clock after meeting the initial calibration acceptance criteria, samples may be analyzed. The calibration must be verified each twelve hour time period (time period starts from the

moment of the DFTPP injection) for Method 8270-SIM. The SSTD1.0 in the curve may be used as the continuing calibration standard as long as it meets the continuing calibration acceptance criteria. All sample results must be quantitated using the initial calibration response factors.

7.5.2.2 Immediately following calibration an Independent Calibration Verification Standard must be analyzed. For clients requiring DOD criteria, all project analytes must be within +/- 20% of true value.

7.5.3 Continuing Calibration

A check of the calibration curve must be performed once every twelve hours immediately following analysis of the tuning compound DFTPP. This check contains all target compounds and surrogates at a concentration of 1.0 ng/uL.

After quantitation of the 1.0 ng/uL continuing calibration check, response factors must be calculated and compared to the average response factors in the initial calibration. The Target program calculates the calibration check response factors and compares them to the average RFs in the calibration curve by calculating percent differences. The method 8270 CCC's must have a % difference of +/- 20%D in order to be considered in criteria. These conditions must be met before method blank and/or sample analysis can begin. For clients requiring DOD criteria, all project analytes and surrogates must be within +/- 20%.

If the continuing calibration check does not meet criteria, corrective action must be taken. Depending on the situation, corrective action may be as follows:

- Re-analyze the 1.0 ng/uL continuing calibration check.
- Change the septum; clean the injection port; install a clean, silanized quartz liner; cut off a small portion (1" to 3") of the front end of the capillary column. This is usually performed when chromatography is poor. Record any of these actions in the appropriate instrument maintenance logbook.

- Analyze a new initial calibration curve.

The last option, the generation of a new initial calibration curve, is usually chosen when percent difference are >30%. In these instances, there is little or no chance of a continuing calibration reanalysis meeting criteria. If there is any doubt concerning which corrective action to undertake, consult the Organic Department Manager or a senior chemist within the group.

If the continuing calibration does meet the criteria specified above then analysis may proceed using initial calibration response factors.

7.5.4. Retention Time Windows

Retention time windows are set at the midpoint standard of the calibration curve, following every ICAL. When a CV is analyzed (and not an ICAL), the retention time windows of the daily CV must be within 30 seconds of the midpoint calibration standard of the most recent ICAL. The samples analyzed following the daily CV must have retention times within 30 seconds of those for the daily CV. Each successive daily CV must be compared to the most recent ICAL midpoint standard.

7.6 SAMPLE ANALYSIS

Sample extracts may be analyzed only after the GC/MS system has met tuning criteria, initial calibration and continuing calibration requirements. Ensure that the same instrument conditions are being used for tuning, calibration and sample analysis by reviewing the GC parameters using the "Edit entire method" option under the Method menu in MSTOP. Note that you can not edit a method if the instrument is running.

Extracts are stored in the refrigerator in the organics extraction laboratory at 4°C ±2°C. Remove them from the refrigerator and place them in the GC/MS laboratory semivolatile hood when ready for analysis.

Prepare a 1.8 mL clear glass vial (crimp top) with a disposable insert (350 uL). Add 100 uL of sample extract and 1.0 uL of the 80 ng/uL IS stock to the vial and then cap. This gives a 0.8 ng/uL final concentration for the internal standard compounds. The samples are topped with Teflon lined crimp top caps.

7.7 FINAL DATA PACKAGE

7.7.1 Initial Data Review (IDR)

The initial data review is accomplished by the analyst who ran the samples and is a review of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed. The initial data review is performed on the detailed quantitation reports of the analyzed sample. This data review examines criteria that directly impact whether or not the sample needs to be reanalyzed:

- Surrogate Recoveries
- Internal Standard Area Stability

- Method Blank Acceptance
- Chromatography
- Target Compound Detection/Quantitation/Review for false positives
- Laboratory Control Sample Recoveries
- Matrix Spike/Matrix Spike Duplicate Recoveries

The analyst must evaluate all data using the QA Acceptance Criteria table found within this SOP (Table 1). This table gives acceptance criteria and corrective actions for criteria that are not met. In addition to evaluating QC elements, the chromatography and quantitation of target analytes must be reviewed. During this review, the analyst checks the integration of each individual peak. The hardcopy has false positives crossed out so they can be reviewed for appropriateness by the Organic Department Manager.

7.7.2 Chromatography

The chromatography should be examined for the presence or absence of any ghost peaks and can also be used as an indication of whether or not matrix interferences might be affecting surrogate recoveries and/or istd area recoveries. Whether or not the chromatography is acceptable is a judgment call on the part of the analyst and should be used in conjunction with other monitored QC (e.g. surrogate recoveries) to determine the necessity of reanalyzing.

Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating the peak of concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.

Each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. Should a manual integration be necessary (for instance, due to a split peak, peak tailing, or incomplete resolution of isomeric pairs), an “m” qualifier will automatically be printed on the quantitation report summary.

This manual integration package must then be submitted to the Department Manager or his/her designee, who will review each manual integration.

For specific manual integration procedures, refer to Katahdin SOP QA-812, “Manual Integration”, current revision.

7.7.3 Target Compound Detection/Quantitation

The semivolatile ID files have been set up to err on the side of false positives; that is to identify and quantitate peaks as target compounds that may not necessarily be valid hits. It is the responsibility of the GC/MS analyst to use his/her technical judgment to determine if the identification of a target compound is correct or not.

If any target concentration exceeds the upper limit, a dilution must be made and analyzed. The dilution chosen should keep the concentration of the largest target compound hit in the upper half of the initial calibration range. LCS and MS/MSD samples need not be diluted to get spiked analytes within the calibration range.

The requirements for qualitative verification by comparison of mass spectra are as follows:

- All ions present in the standard mass spectra at a relative intensity > 10% must be present in the sample spectrum.
- The relative intensities of primary and secondary ions must agree within ±20% between the standard and sample spectra.
- Ions greater than 10% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst.

If a compound cannot be verified by all three criteria above, but, in the technical judgment of the mass spectral interpretation specialist, the identification is correct, then the laboratory shall report that compound on the Form 1 as a valid hit.

The GC/MS laboratory initial data review must be completed within twelve hours of batch completion; in the majority of instances, the initial review should be accomplished at the beginning of a work shift for the previous set of analyses.

7.7.4 Reporting

After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into QuickForms. Depending on the QC label requested by the client, a Report of Analysis (ROA) and additional reports, such as LCS forms and chronology forms, are generated. The package is assembled to include the necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded to the secondary reviewer. The secondary reviewer validates the data and checks the package for any errors. When completed, the package is sent to the department manager for final review. A complete review checklist is provided with each package. The final data package from

the Organics department is then processed by the Data Management department.

7.8 INJECTION PORT LINER CLEANING AND SILANIZING PROCEDURE

- Remove the rubber o-ring from the liner and place the liner in a large Erlenmeyer flask.
- In the hood, pour nitric acid into the flask until the liner is covered. Place the flask on a hotplate and boil for 2-3 hours.
- Let cool; drain nitric acid and thoroughly flush the liner with water.
- Bake briefly in the muffle oven until liner is dry and cool to room temperature.
- Place the liner in a beaker, fill with Sylon and let it soak for at least two hours.
- Take out the liner and rinse it thoroughly with toluene.
- Rinse the liner thoroughly with purge and trap grade methanol.
- Bake the liner in the muffle oven for a minimum of three hours.

7.9 Instrument Maintenance

Instrument preventative maintenance is performed on a semi-annual basis by GC/MS chemists. This maintenance includes a thorough inspection and cleaning of all parts, including changing rough and turbopump oils. GC/MS analysts perform other maintenance on an as-needed basis. Typically, routine maintenance involves clipping off the front end of the DB-5MS column, replacing the injection port septum, and installing a freshly silanized quartz liner after sample analysis.

All maintenance must be documented in the instrument-specific maintenance log, whether it is routine or not. The Department Manager must authorize any maintenance over and above a routine source cleaning.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 and to details in this section for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The criteria does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in this section or in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in this section and in Table 1 may rely on analyst experience to make sound scientific judgements. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases “qualified” data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 Method Blank Criteria

A method blank is defined as a volume of a clean reference material (deionized distilled water for water samples, baked organic-free sand for soil/sediment matrices) that is carried through the entire analytical procedure. One method blank must be extracted with each group of samples of a similar matrix and must be analyzed on the GC/MS system that was used to analyze the samples.
An acceptable method blank must contain less than or equal to the PQL of any target compound. For clients requiring DOD criteria, no analytes detected at > ½ PQL and > 1/10 the amount measrued in any sample or 1/10 the regulatory limit.

If the method blank exceeds these contamination levels, the analytical system is considered out of control and corrective action must be taken before sample analysis.

Reanalysis of the blank is the first step of the corrective action; if that does not solve the problem, a Katahdin Corrective Action Report (CAR) will be initiated. Corrective action will be specified after consultation including the Department Manager, Operations Manager, and QA Officer.

8.2 Surrogate Recoveries

The four surrogates (2-Methylnaphthalene-d10, 2,4-Dibromophenol, Fluorene-d10 and Pyrene-d10) must meet the current statistically derived acceptance limits. If statistical limits have not been established then the surrogate recovery must meet the nominal limits of 30-150%. For clients requiring DOD criteria, use acceptance limits specified by DOD or use in-house limits where none are specified.

If specifications are not met, the sample (or blank) should be reanalyzed. If specifications are met in the reanalysis, this reanalysis should only be submitted. If surrogate specifications are not met in the sample or method blank reanalysis, a Corrective Action Report (CAR) should be initiated. Corrective action will be specified after consultation including the Department Manager and Operations Manager.

For further information regarding the acceptance of surrogate recoveries, consult the Organic Department Manager.

8.3 Internal Standard Responses

Internal standard responses and retention times (RT) in all samples and blanks must be evaluated as part of the technical data review. The method files have been set up to only detect compounds that fall within a set RT window. For Method 8270-SIM analysis, if the extracted ion current profile (EICP) area for any internal standard changes by more than a factor of two (-50% to +100%) as compared to the daily continuing calibration standard, reanalysis must occur. If the reanalysis meets criteria, only the in-criteria run should be reported. If the reanalysis is still out of criteria, both analyses should be included in the sample package set.
MS/MSD samples that do not meet the EICP area criteria above do not have to be reanalyzed.

8.4 Laboratory Control Sample (LCS)

An LCS must be performed for each group of samples of a similar matrix, for the following, whichever is more frequent:

- Every 20 samples of a similar matrix or similar concentration, or
- Every batch of samples extracted.

Statistical limits are compiled annually for LCS recoveries (archived in QA office). Statistical limits are only calculated when at least 20 usable data points are obtained for any given compound. If insufficient data points are available, nominal limits are set by the section supervisor, Laboratory Operations Manager and Quality Assurance Officer. Refer to Katahdin SOP QA-808, "Generation and Implementation of Statistical QC Limits and/or Control Charts", current revision.

The use of statistical limits versus nominal limits is dependent on the client and project. This information is communicated to the Organic Department Manager through the Katahdin project manager. It is standard practice to use statistical limits for reporting purposes and to evaluate any QC criteria exceedances. However, nominal limits of 30-150% may be used for some projects or states (i.e. South Carolina). For clients requiring DOD criteria, use acceptance limits specified by DOD or use in-house limits where none are specified.

The LCS recoveries for all analytes are evaluated. All of the compounds of interest must fall within the established statistical limits with the following sporadic exceedance allowances.

# of Analytes	# of Allowable Exceedances
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2

# of Analytes	# of Allowable Exceedances
11 – 30	1
<11	0

If less than the number of allowable exceedances fail the statistical limits, no corrective action is needed. If greater than the number of allowable exceedances fail the statistical limits, corrective action may be taken. Corrective actions may vary with each situation. However, in the case where the failures are high and the samples are non-detect for those compounds, then no corrective action is required. Otherwise, corrective action may involve reanalysis or recalibration. The specific corrective actions taken will rely on analyst experience to make sound scientific judgments while considering client objectives, other quality control indicators and/or the ability to reanalyze a sample within holding time

Please note that for compounds with only nominal limits (i.e. insufficient data points were available to generate statistical limits), no corrective action is required for out-of-criteria recoveries until enough data points are established to generate statistical limits.

8.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Criteria

Matrix Spike and Matrix Spike Duplicates must be extracted and analyzed for each group of up to 20 samples of a similar matrix or similar concentration. In the event insufficient sample volume is available an LCS/LCS Duplicate is extracted and analyzed in place of the MS/MSD.

Statistical limits are compiled annually for MS/MSD recoveries for a short list of the spiked compounds (Acenaphthene, Pentachlorophenol and Pyrene). Nominal limits of 30-130% are used for all other compounds. Generally, corrective action is only taken for the short list of the spiked compounds. The specific corrective actions will rely on analyst experience to make sound scientific judgments while considering client objectives, other quality control indicators and/or the ability to reanalyze a sample within holding time. For clients requiring DOD criteria, use acceptance limits specified by DOD or use in-house limits where none are specified.

A Corrective Action Report (CAR) must be filled out and filed if any criteria for percent recovery or relative percent difference are not met to document any decisions with reporting data.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Organic Department

Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revision of Method 8270 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

"Test Methods for the Evaluation of Solid Waste: Physical/Chemical Methods", SW-846, third Edition, Final Updates I, II, IIA, III, IIIA and IIIB, November 2004, Method 8270C.

Department of Defense Quality Systems Manual for Environmental Laboratories (DoD QSM), Version 4.1, 04/22/09

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003

"USEPA Contract Laboratory Program Statement of Work for Organics Analysis," Rev. 02/88.

Code of Federal Regulations (40 CFR), Part 136, Appendix A, Rev. June, 1998.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

LIST OF TABLES AND FIGURES

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 1

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Check of mass spectral ion intensities using DFTPP	Prior to initial calibration and calibration verification	Refer to the criteria listed in Section 7.4	Retune instrument, and verify
Six-point initial calibration for all analytes	Initial calibration prior to sample analysis	RSD ≤30 for RFs of the CCCs; Average %RSD < 15% for all compounds. Refer to section 7.5.2.1 for more details.	Repeat calibration if criterion is not met
Independent calibration verification	Once after Initial calibration	± 20 % D	1) Reanalyze standard 2) Reprep standard 3) Reprep standard from fresh stock.
Continuing calibration verification	Once per each 12 hours, prior to sample analysis	CCCs ≤ 20%D	Repeat initial calibration and reanalyze all samples analyzed since the last successful calibration verification
ISs	Immediately after or during data acquisition of calibration check standard	Retention time ± 30 seconds; EICP area within -50% to +100% of last calibration verification (12 hours) for each IS	Inspect mass spectrometer or GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning
Demonstration of ability to generate acceptable accuracy and precision	Once per analyst initially and annually thereafter	All recoveries within method QC acceptance limits.	Recalculate results; locate and fix problem; reextract/reanalyze P&A study for those analytes that did not meet criteria
Method blank	One per prep batch	No analytes detected > PQL	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e.If the blank results are above the PQL, report samples that are <PQL or > 10X the blank result. Reprep a blank and the remaining samples.
LCS for all analytes	One LCS per prep batch	Statistically derived from lab data or nominal limits depending on the project. . See also section 8.4 of this SOP for more information on allowable exceedances.	(1) Evaluate the samples and associated QC: i.e.If an MS/MSD was performed and acceptable, narrate. If an LCS/LCSD was performed and only one was unacceptable, narrate. If the surrogate recoveries in the LCS are low but are acceptable in the blank and samples, narrate. If the LCS rec. is high but the sample results are <PQL, narrate. Otherwise, reprep a blank and the remaining samples.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 1

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Surrogate spike	Every sample, control, standard, and method blank	Statistically derived limits.	(1) Check chromatogram for interference; if found, flag data (2) If not found, check instrument performance; if problem is found, correct and reanalyze (3) If still out reextract and analyze sample (4) If reanalysis is out, flag data
MS/MSD	One MS/MSD per every 20 samples	Statistically derived from lab data or nominal limits depending on the project. Nominal limits are used as default limits.	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS/MSD are unacceptable reprep the samples and QC.
MDL and/or LOD/LOQ Verification study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 2

DoD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specific criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria.	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification	Refer to current revision of SOP QA-806				
LOQ establishment and verification	Refer to current revision of SOP QA-806				
Tuning	Prior to ICAL and at the beginning of each 12-hour period.	Refer to method for specific ion criteria.	Retune instrument and verify. Rerun affected samples.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be accepted without a valid tune.
Breakdown check (DDT Method 8270 only)	Correct problem then repeat breakdown check.	Degradation ≤ 20% for DDT. Benzidine and pentachlorophenol should be present at their normal responses, and should not exceed a tailing factor of 2.	At the beginning of each 12-hour period, prior to analysis of samples.	Flagging criteria are not appropriate.	No samples shall be run until degradation ≤ 20%.
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	1. Average response factor (RF) for SPCCs ≥ 0.050. 2. RSD for RFs for CCCs ≤ 30% and one option below: Option 1: RSD for each analyte ≤ 15%; Option 2: linear least squares regression r ≥ 0.995; Option 3: non-linear regression– coefficient of determination (COD) r2 ≥ 0.99 (6 points shall be used for second order).	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. Calibration may not be forced through the origin.
Second source calibration verification (ICV)	Once after each ICAL.	All project analytes within ± 20% of true value.	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.

TABLE 2 (cont)

DoD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Retention time window position establishment for each analyte and surrogate	Once per ICAL.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	
Evaluation of relative retention times (RRT)	With each sample.	RRT of each target analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	Flagging criteria are not appropriate.	Laboratories may update the retention times based on the CCV to account for minor performance fluctuations or after routine system maintenance (such as column clipping). With each sample, the RRT shall be compared with the most recently updated RRT. If the RRT has changed by more than ±0.06 RRT units since the last update, this indicates a significant change in system performance and the laboratory must take appropriate corrective actions as required by the method and rerun the ICAL to reestablish the retention times.
Continuing calibration verification (CCV)	Daily before sample analysis and every 12 hours of analysis time.	1. Average RF for SPCCs ≥ 0.050. 2. %Difference/Drift for all target compounds and surrogates ≤ 20%D (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration).	DoD project level approval must be obtained for each of the failed analytes or corrective action must be taken. Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since last acceptable CCV.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Internal standards verification	Every field sample, standard, and QC sample.	Retention time ± 30 seconds from retention time of the midpoint standard in the ICAL; EICP area within -50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, apply Q-flag to analytes associated with the non-compliant IS. Flagging criteria are not appropriate for failed standards.	Sample results are not acceptable without a valid IS verification.

TABLE 2 (cont)

DoD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method blank	One per preparatory batch.	No analytes detected > ½ RL (> RL for common lab contaminants) and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Reprepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result. Contact Client if samples cannot be reprepped within hold time.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
LCS containing all analytes to be reported, including surrogates	One per preparatory batch.	The laboratory shall use laboratory control limits (CLs) or use DoD-generated LCS-CLs, if available depending on project requirements. In-house CLs may not be greater than ± 3 times the standard deviation of the mean LCS recovery. A number of analytes may fall outside the CL but within marginal exceedance limit depending on the total number of analytes in the LCS.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. Refer to Table G-1 for number of marginal exceedences allowed. Contact Client if samples cannot be reprepped within hold time.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch per matrix if sufficient sample is available.	For matrix evaluation, use LCS acceptance criteria.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix if sufficient sample is available.	MSD: For matrix evaluation, use LCS acceptance criteria. MS/MSD: RPD ≤ 30%.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.

TABLE 2 (cont)

DoD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Surrogate spike	All field and QC samples.	The laboratory shall use laboratory surrogate CLs or use DoD-generated surrogate CLs, if available depending on project requirements. .	For QC and field samples, correct problem then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary. Contact Client if samples cannot be reprepped within hold time.	Apply Q-flag to all associated analytes if acceptance criteria are not met.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

TABLE 3

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-213-08	METHOD 8270, current revision
Apparatus/Materials	none	
Reagents	none	
Sample preservation/ handling	none	
Procedures	none	
QC - Spikes	none	
QC - LCS	none	
QC - Accuracy/Precision	none	
QC - MDL	none	

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 4

ANALYTE QUANITIATION AND INTERNAL STANDARDS

Internal Standard: 1,4-dichlorobenzene-d4
Target and Surrogates:
1,4-Dioxane
Benzaldehyde
Phenol
bis(2-Chloroethyl)ether
2-Chlorophenol
2-Methylphenol
3&4-Methylphenol
2,2'-Oxybis(1-chloropropane)
Nitrobenzene
Hexachloroethane
Acetophenone
N-nitroso-di-n-propylamine
Internal Standard: Naphthalene-d8
Target and Surrogates:
Naphthalene
1-Methylnaphthalene (dredge)
2-Methylnaphthalene
2-Methylnaphthalene-D10 (surrogate)
Isophorone
2-Nitrophenol
2,4-Dimethylphenol
bis(2-Chloroethoxy)methane
2,4-Dichlorophenol
4-Chloroaniline
Hexachlorobutadiene
Caprolactam
4-Chloro-3-methylphenol
Internal Standard: Acenaphthene-d10
Target and Surrogates:
1,1'-Biphenyl (dredge)
2,6 Dimethylnapthalene (dredge)
Acenaphthylene
Acenaphthene
Fluorene
2-Fluorene-d10 (surrogate)
2,4-Dibromophenol (surrogate)
2-Chloronaphthalene
Hexachlorocyclopentadiene
2,4,6-Trichlorophenol
2,4,5-Trichlorophenol
Dimethylphthalate
2,6-Dinitrotoluene

2,4-Dinitrotoluene
2,4-Dinitrophenol
2,3,4,6-Tetrachlorophenol
Diethylphthalate
4-Chlorophenyl-phenyl ether
4,6-Dinitro-2-methylphenol
N-nitrosodiphenylamine
2-Nitroaniline
3-Nitroaniline
4-Nitroaniline
Dibenzofuran
4-Nitrophenol
Internal Standard: Phenanthrene-d10
Target and Surrogates:
Pentachlorophenol
1-Methylphenanthrene (dredge)
Phenanthrene
Hexachlorobenzene (special)
Anthracene
Fluoranthene
Carbazole
Di-n-butylphthalate
4-Bromophenyl-phenyl ether
Atrazine
Internal Standard: Chrysene-d12
Target and Surrogates:
Butylbenzylphthalate
3,3'-Dichlorobenzidine
Pyrene
Benzo(a)Anthracene
Chrysene
Bis-(2-ethylhexyl)phthalate
Pyrene-d10 (surrogate)
Internal Standard: Perylene-d12
Target and Surrogates:
Perylene (dredge)
Benzo(b)fluoranthene
Benzo(k)fluoranthene
Benzo(e)pyrene (dredge)
Di-n-octylphthalate
Benzo(a)pyrene
Indeno(1,2,3-cd)pyrene
Dibenz(a,h)anthracene
Benzo(ghi)perylene

TABLE 5

PROCEDURE CONDENSATION

Clock

12 hours from injection of 50ng DFTPP.

Calibration Curve Criteria

<30% RSD for CCCS
<15% RSD average for all analytes in calibration standard

Continuing Calibration Check Criteria

<20% D for CCC compounds

Additional QC

LCS every extraction batch
MS/MSD every 20 samples

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 6

SVOA COMPOUNDS AND CHARACERISTIC IONS

COMPOUND	PRIMARY ION	SECONDARY IONS
1,4-Dioxane	88	58
Benzaldehyde	77	105,106
Phenol	94	65,66
bis(2-Chloroethyl)ether	93	63,95
2-Chlorophenol	128	64,130
1,4-Dichlorobenzene-d4 (IS)	152	150,115
2,2'-Oxybis(1-choropropane)	45	77,121
2-Methylphenol	108	107,77
Acetophenone	105	77,51
N-nitroso-di-n-propylamine	70	52,101
Hexachloroethane	117	201,199
3&4-Methylphenol	108	107,77
Nitrobenzene	77	123,51
Isophorone	82	54,138
2-Nitrophenol	139	109,81
2,4-Dimethylphenol	107	122,121
bis(2-Chloroethoxy)methane	93	63,123
2,4-Dichlorophenol	162	164,98
Naphthalene-d8 (IS)	136	137,134
Naphthalene	128	129,127
4-Chloroaniline	127	129
Hexachlorobutadiene	225	223,227
Caprolactam	113	55,56
4-Chloro-3-methylphenol	107	77,142
2,4-Dibromophenol (surr)	252	63,143
2-Methylnaphthalene-d10 (surr)	152	150
2-Methylnaphthalene	142	141,115
1-Methylnaphthalene	142	141,115
Hexachlorocyclopentadiene	237	235,239
2,4,6-Trichlorophenol	196	198,200
2,4,5-Trichlorophenol	196	198,200
2-Chloronaphthalene	162	127,164
1,1'-Biphenyl	154	153,76
2-Nitroaniline	65	92,138
Dimethylphthalate	163	194,164
2,6-Dinitrotoluene	165	63,89
Acenaphthylene	152	151,153
Acenaphthene	152	154,152
Acenaphthene-d10 (IS)	164	162
3-Nitroaniline	138	65,92
2,4-Dinitrophenol	184	107
Dibenzofuran	168	139

TABLE 6 (cont.)

SVOA COMPOUNDS AND CHARACERISTIC IONS

COMPOUND	PRIMARY ION	SECONDARY IONS
2,4-Dinitrotoluene	165	63
4-Nitrophenol	109	139,65
2,3,4,6-Ttrachlorophenol	232	230
Diethylphthalate	149	177,176
Fluorene-d10 (surr)	176	174,178
Fluorene	166	165
4-Chlorophenyl-phenyl ether	204	206,141
4-Nitroaniline	138	108,65
4,6-Dinitro-2-methylphenol	198	121
N-nitrosodiphenylamine	169	168,167
4-Bromophenyl-phenyl ether	248	250,141
Hexachlorobenzene	284	142,249
Atrazine	200	173,215
Pentachlorophenol	266	264,268
Phenanthrene-d10 (IS)	188	189
Phenanthrene	178	179,176
Anthracene	178	179,176
Carbazole	167	166,139
Di-n-butylphthalate	149	150,104
Fluoranthene	202	200,203
Pyrene	202	200,201
Pyrene-d10 (surr)	212	210,106
Butylbenzylphthalate	149	91,206
Benzo(a)anthracene	228	229,226
Chrysene-d12 (IS)	240	236,120
3,3-Dichlorobenzidine	252	254,126
Chrysene	228	226,229
bis(2-Ethylhexyl)phthalate	149	167
Di-n-octylphthalate	149	150
Benzo(b)fluoranthene	252	253,125
Benzo(k)fluoranthene	252	253,125
Benzo(a)pyrene	252	253,250
Perylene-d12 (IS)	264	260
Indeno(1,2,3-cd)pyrene	276	277
Dibenzo(a,h)anthracene	278	279
Benzo(g,h,i)perylene	276	277

Primary ions must not be changed except in unusual instances where interference occurs with a co-eluting non-target analyte. In this case, a secondary ion may be used for quantitation with the following rules:
(1) The corresponding standard(s) (initial calibration curve and continuing calibration standard) must be re-quantitated with the secondary ion.

TABLE 6 (cont.)

SVOA COMPOUNDS AND CHARACERISTIC IONS

(2) Approval must be obtained from the Organic Department Manager or the laboratory Operations Manager.

The quantitation ion must then be changed back to the one specified in the table above after quantitation of the samples(s).

Secondary ions are recommended only and may be changed depending upon instrument conditions (sensitivity, etc.). However, it is Katahdin policy that a minimum of 2 ions (primary and one secondary) be used for all GC/MS analyses.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

FIGURE 1

EXAMPLE OF RUNLOG LOGBOOK PAGE

KATAHDIN ANALYTICAL SERVICES
GC/MS SVOA INJ LOG INSTRUMENT: 5970-X

DATE OF DFTPP INJECTION: 03/0C9

JOB	SAMPLE	DATAFILE	DF	ALS #	METHOD	UL INJ	CHEMIST	COMMENTS
	50ng DFTPP	X0600	1	1	DFTPP390	2.0	SLR	OK
	SSTD060X0316	X9437		2	X625A022	1.0		✓
	150	38		3				✓
	120	39		4				✓ curve OK
	30	40		5				✓
	05	41		6				✓
	3510 MIX 1	42		7				OK
	2	43		8				
	3	44		9				
	4	45		10				
	5	46		11				
	6	47		12				
	7	48		13				
	8	49		14				
	9	50		15				
	BLANK 1	51		16				
	2	52		17				

STANDARD	CODE
DFTPP	50856
CAL. STD.	5089-30 5089-49
IS MIX	AMP0934

REVIEWED AND APPROVED BY: _____
DATE: _____

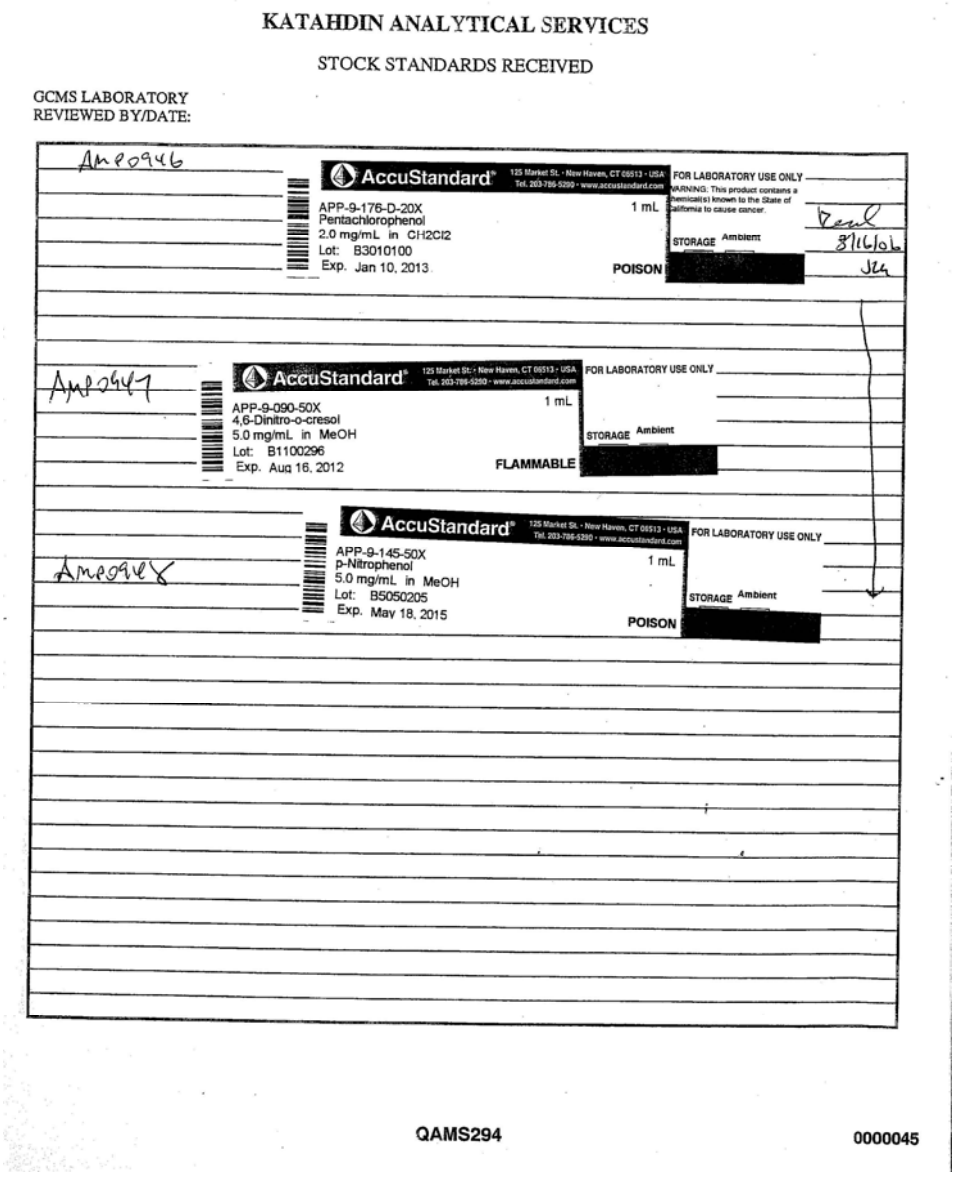
QAMS302

0000034

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

FIGURE 2

EXAMPLE OF GC/MS STANDARDS RECEIPT LOGBOOK ENTRY



KATAHDIN ANALYTICAL SERVICES

STOCK STANDARDS RECEIVED

GCMS LABORATORY
REVIEWED BY/DATE:

Amp0946

AccuStandard® 125 Market St. · New Haven, CT 06513 · USA Tel. 203-786-5290 · www.accustandard.com

APP-9-176-D-20X
Pentachlorophenol
2.0 mg/mL in CH2Cl2
Lot: B3010100
Exp. Jan 10, 2013

1 mL

FOR LABORATORY USE ONLY
WARNING: This product contains a chemical(s) known to the State of California to cause cancer.

STORAGE Ambient

POISON [redacted]

Dml 8/16/06 JLH

Amp0947

AccuStandard® 125 Market St. · New Haven, CT 06513 · USA Tel. 203-786-5290 · www.accustandard.com

APP-9-090-50X
4,6-Dinitro-o-cresol
5.0 mg/mL in MeOH
Lot: B1100296
Exp. Aug 16, 2012

1 mL

FOR LABORATORY USE ONLY

STORAGE Ambient

FLAMMABLE [redacted]

Amp0948

AccuStandard® 125 Market St. · New Haven, CT 06513 · USA Tel. 203-786-5290 · www.accustandard.com

APP-9-145-50X
p-Nitrophenol
5.0 mg/mL in MeOH
Lot: B5050205
Exp. May 18, 2015

1 mL

FOR LABORATORY USE ONLY

STORAGE Ambient

POISON [redacted]

QAMS294

0000045

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

FIGURE 3

EXAMPLE OF SVOA STANDARDS PREPARATION LOGBOOK ENTRY

GC/MS SVOA STANDARD PREP LOGBOOK

50863	8270 Stock (w/o MeOH)	3-15-06	7-7-06	JLh	AMP0844	8270 Megamix	300	2-22-07	4.2ml	150 ug/ml
					AMP0937	↓	350	3-15-07		
					AMP0911	APP IX #2	600	3-2-07		
					AMP0910	↓ 1	180	3-9-07		
					AMP0870	↓ 1	200	7-7-06		
					AMP0699	Organophosphat	300	5-14-06		
					AMP0938	Benzoic Acid	↓	3-9-07		
					AMP0907	Hexachlorophene		2-22-07		
					AMP0806	Benzidine		3-9-07		
					AMP0936	3,3'-Dichlorobenzidine	↓	3-14-07		
					AMP0932	8270 Surr	150	3-9-07		
					50861	DEA	300	3-13-07		
					BY3E90	MeCl2	550	~		
50864	8270 level 1	3-15-06	7-7-06	JLh	50863	8270 Stock	70	7-7-06	1.05ml	10 ug/ml
					BY3E90	MeCl2	980			
50865	8270 level 2	3-15-06	7-7-06	JLh	50863	8270 Stock	150	7-7-06	0.90ml	25 ug/ml
					BY3E90	MeCl2	750			
50866	8270 level 3	3-15-06	7-7-06	JLh	50863	8270 Stock	600	7-7-06	1.8ml	50 ug/ml
					BY3E90	MeCl2	1200			
50867	8270 level 4	3-15-06	7-7-06	JLh	50864	8270 Stock	700	7-7-06	1.05ml	100 ug/ml
					BY3E90	MeCl2	350			

Reviewed by/Date:

CAMS284

0000057

TITLE: CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035

Prepared By: GC/MS Group Date: 7/98

Approved By:

Group Supervisor: [signature] Date: 011201

Operations Manager: John C. Burton Date: 1/15/01

QA Officer: Deborah J. Nadeau Date: 1/23/01

General Manager: [signature] Date: 1/16/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 5035	Format changes, added pollution prevention, minor changes throughout	DN	1/23/01	1/30/01
02 5035	Reorganized sections 4, 5, 6, 7 and 8.	MRC	07.02.04	07.02.04
03 5035	Editted section 6.4.3 to include the addition of 5mL of H2O to sample	LAD	020305	020305
04 5035	Balance weighs to 0.1g grammatical corrections formatting corrections	LAD	04/06	04/06

TITLE: **CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-214-04**, titled **CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-214-04**, titled **CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035.**

Recipient: _____ Date: _____

TITLE: **CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035**

1.0 SCOPE AND APPLICATION

This method describes a closed-system purge-and-trap process for the analysis of volatile organic compounds (VOCs) in solid materials (e.g., soils, sediments, and solid waste). While the method is designed for use on samples containing low levels of VOCs, procedures are also provided for collecting and preparing solid samples containing high concentrations of VOCs and for oily wastes. For these high concentration and oily materials, sample collection and preparation are performed using the procedures described here, and sample introduction is performed using the aqueous purge-and-trap procedure in Method 5030. These procedures may be used in conjunction with any appropriate determinative gas chromatographic procedure, including, but not limited to, Methods 8015, 8021, and 8260.

The low soil method utilizes a hermetically sealed sample vial, the seal of which is never broken from the time of sampling to the time of analysis. Since the sample is never exposed to the atmosphere after sampling, the losses of VOCs during sample transport, handling, and analysis are negligible. The applicable concentration range of the low soil method is dependent on the determinative method, matrix, and compound. However, it will generally fall in the 5.0 to 200 µg/kg range.

Procedures are included for preparing high concentration samples for purging by Method 5030. High concentration samples are those containing VOC levels of >200 µg/kg.

Procedures are also included for addressing oily wastes that are soluble in a water-miscible solvent. These samples are also purged using Method 5030.

Method 5035 can be used for most volatile organic compounds that have boiling points below 200°C and that are insoluble or slightly soluble in water. Volatile, water-soluble compounds can be included in this analytical technique. However, quantitation limits (by GC or GC/MS) are approximately ten times higher because of poor purging efficiency.

The closed-system purge-and-trap equipment employed for low concentration samples is not appropriate for soil samples preserved in the field with methanol. Such samples should be analyzed using Method 5030 (see the note in Sec. 6.2.2).

1.1 Definitions

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analyses using Methods 8015, 8021, 8260 and CLP. Each analyst must demonstrate and document their ability to generate acceptable results with this

method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Demonstration of Capability".

It is the responsibility of all Katahdin technical personnel involved in analysis of soils by method 5035 to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the department manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures and the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of a respirator and all safety equipment. Each analyst shall receive a safety orientation from their department manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

After analysis, partially-filled VOA vials and sample jars are returned to the appropriate refrigerators to be disposed of in adherence with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, Sample Disposal, current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP SD-903.

Sample aliquots used for analysis are disposed of in accordance with SOP SD-903 and the Katahdin Hazardous Waste Management Plan and Safety Manual. The soil samples must be decanted and the soil fraction disposed of separately in compliance with Katahdin's disposal policies.

2.0 SUMMARY OF METHOD

2.1 Low concentration soil method - generally applicable to and soils and other solid samples with VOC concentrations in the range of 5.0 to 200 µg/kg. Volatile organic compounds (VOCs) are determined by collecting an approximately 5-g sample, weighed in the field at the time of collection, and placing it in a pre-weighed vial with a septum-sealed screw-cap (see Sec. 4) that already contains a stirring bar and a sodium bisulfate or organic-free laboratory reagent grade water preservative solution. If the samples are sent to the laboratory in an Encore sampling device, the laboratory extrudes the sample into this vial containing a stirring bar and a sodium bisulfate or organic-free laboratory reagent grade water preservative solution. The entire vial is then placed, unopened, into the instrument carousel. Immediately before analysis, organic-free laboratory reagent grade water, surrogates, and internal standards (if applicable) are automatically added without opening the sample vial. The vial containing the sample is heated to 40° and the volatiles purged into an appropriate trap using an inert gas combined with agitation of the sample. Purged components travel via a transfer line to a trap. When purging is complete, the trap is heated and backflushed with helium to desorb the trapped sample components into a gas chromatograph for analysis by an appropriate determinative method.

2.2 High concentration soil method - generally applicable to soils and other solid samples with VOC concentrations greater than 200 µg/kg. The sample introduction technique in Sec. 2.1 is not applicable to all samples, particularly those containing high concentrations (generally greater than 200 µg/kg) of VOCs which may overload either the volatile trapping material or exceed the working range of the determinative instrument system (e.g., GC/MS, GC/FID, GC/EC, etc.). In such

instances, this method describes two sample collection options and the corresponding sample purging procedures.

2.2.1 The first option is to collect a bulk sample in a vial or other suitable container without the use of the preservative solution described in Sec. 2.1. A portion of that sample is removed from the container in the laboratory and is dispersed in a water-miscible solvent (e.g., methanol) to dissolve the volatile organic constituents. An aliquot of the solution is added to 20 mL of laboratory reagent grade water in a purge tube. Surrogates and internal standards (if applicable) are added to the solution, then purged using Method 5030, and analyzed by an appropriate determinative method. Because the procedure involves opening the vial and removing a portion of the soil, some volatile constituents may be lost during handling.

2.2.2 The second option is to collect an approximately 5-g sample in a pre-weighed vial with a septum-sealed screw-cap (see Sec 4) that contains a known aliquot of a water-miscible organic solvent (e.g., methanol). An aliquot of the solution is added to 20 mL of laboratory reagent grade water in a purge tube. Surrogates and internal standards (if applicable) are added to the solution, then purged using Method 5030, and analyzed by an appropriate determinative method.

2.3 High concentration oily waste method - generally applicable to oily samples with VOC concentrations greater than 200 µg/kg that can be diluted in a water-miscible solvent. Samples that are comprised of oils or samples that contain significant amounts of oil present additional analytical challenges. This procedure is generally appropriate for such samples when they are soluble in a water-miscible solvent.

2.3.1 After demonstrating that a test aliquot of the sample is soluble in methanol, a separate aliquot of the sample is diluted in the appropriate solvent. An aliquot of the solution is added to 20 mL of laboratory reagent grade water in a purge tube, taking care to ensure that a floating layer of oil is not present in the purge tube. Internal standards (if applicable) and surrogates are added to the solution that is then purged using Method 5030 and analyzed by an appropriate determinative method.

2.3.2 Samples that contain oily materials that are not soluble in water-miscible solvents must be prepared according to Method 3585.

TITLE: **CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035**

3.0 INTERFERENCES

3.1 Impurities in the purge gas and from organic compounds out-gassing from the plumbing ahead of the trap account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running method blanks. The use of non-polytetrafluoroethylene (non-PTFE) plastic coating, non-PTFE thread sealants, or flow controllers with rubber components in the purging device must be avoided, since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation. These compounds will result in interferences or false positives in the determinative step.

3.2 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample vial during shipment and storage. A trip blank prepared from organic-free laboratory reagent grade water and carried through sampling and handling protocols serves as a check on such contamination.

3.3 Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed in sequence. Where practical, samples with unusually high concentrations of analytes should be followed by an analysis of organic-free laboratory reagent grade water to check for cross-contamination. If the target compounds present in an unusually concentrated sample are also found to be present in the subsequent samples, the analyst must demonstrate that the compounds are not due to carryover. Conversely, if those target compounds are not present in the subsequent sample, then the analysis of organic-free laboratory reagent grade water is not necessary.

3.4 The laboratory where volatile analysis is performed should be free of solvents. The analytical and sample storage area should be isolated from all atmospheric sources of methylene chloride, otherwise random background levels will result. Since methylene chloride will permeate through PTFE tubing, all GC carrier gas lines and purge gas plumbing should be constructed of stainless steel or copper tubing. Laboratory workers' clothing previously exposed to methylene chloride fumes during common liquid/liquid extraction procedures can contribute to sample contamination. The presence of other organic solvents in the laboratory where volatile organics are analyzed will also lead to random background levels and the same precautions must be taken.

TITLE: **CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035**

4.0 APPARATUS AND MATERIALS

4.1 Sample Containers

The specific sample containers required will depend on the purge-and-trap system to be employed (see Sec. 4.2). Our laboratory is equipped with Archon model 5100 purge and trap autosampler systems. A standard 40 ml VOA vial is used (e.g. ESS pre-cleaned certified 40 ml clear Type I borosilicate glass vials, open-top/polypropylene with 0.125 inch septa).

4.2 Purge-and-Trap System

The purge-and-trap system consists of a unit that automatically adds water, surrogates, and internal standards to a vial containing the sample, purges the VOCs using an inert gas stream while agitating the contents of the vial, and also traps the released VOCs for subsequent desorption into the gas chromatograph. The Archon systems at Katahdin meet the following criteria:

4.2.1 The purging device should be capable of accepting a vial sufficiently large to contain a 5-g soil sample plus a magnetic stirring bar and 10 mL of water. The device must be capable of heating a soil vial to 40°C and holding it at that temperature while the inert purge gas is allowed to pass through the sample. The device should also be capable of introducing at least 20 mL of organic-free laboratory reagent grade water into the sample vial while trapping the displaced headspace vapors. It must also be capable of agitating the sealed sample during purging, (e.g., using a magnetic stirring bar added to the vial prior to sample collection, sonication, or other means). The analytes being purged must be quantitatively transferred to an absorber trap. The trap must be capable of transferring the absorbed VOCs to the gas chromatograph (see 4.2.2).

4.2.2 A variety of traps and trapping materials may be employed with this method. The choice of trapping material may depend on the analytes of interest. Whichever trap is employed; it must demonstrate sufficient adsorption and desorption characteristics to meet the quantitation limits of all the target analytes for a given project and the QC requirements in Method 8000 and the determinative method. The most difficult analytes are generally the gases, especially dichlorodifluoromethane. The trap must be capable of desorbing the late eluting target analytes.

NOTE: Check the responses of the brominated compounds when using alternative charcoal traps (especially Vocarb 4000), as some degradation has been noted when higher desorption temperatures (especially above 240°C -

250°C) are employed. 2-Chloroethyl vinyl ether is degraded on Vocarb 4000 but performs adequately when Vocarb 3000 is used. The primary criterion, as stated above, is that all target analytes meet the sensitivity requirements for a given project.

4.2.2.1 The standard trap used in other EPA purge-and-trap methods is also acceptable. That trap is 25 cm long and has an inside diameter of at least 0.105 in. Starting from the inlet, the trap contains the equal amounts of the adsorbents listed below. It is recommended that 1.0 cm of methyl silicone-coated packing (35/60 mesh, Davison, grade 15 or equivalent) be inserted at the inlet to extend the life of the trap. If the analysis of dichlorodifluoromethane or other fluorocarbons of similar volatility is not required, then the charcoal can be eliminated and the polymer increased to fill 2/3 of the trap. If only compounds boiling above 35° are to be analyzed, both the silica gel and charcoal can be eliminated and the polymer increased to fill the entire trap.

4.2.2.1.1 2,6-Diphenylene oxide polymer - 60/80 mesh, chromatographic grade (Tenax GC or equivalent).

4.2.2.1.2 Methyl silicone packing - OV-1 (3%) on Chromosorb-W, 60/80 mesh or equivalent.

4.2.2.1.3 Coconut charcoal - Prepare from Barnebey Cheney, CA-580-26, or equivalent, by crushing through 26 mesh screen.

4.2.2.2 Trapping materials other than those listed above also may be employed, provided that they meet the specifications in Sec. 4.2.3, below.

4.2.3 The desorber for the trap must be capable of rapidly heating the trap to the temperature recommended by the trap material manufacturer, prior to the beginning of the flow of desorption gas. Several commercial desorbers (purge-and-trap units) are available.

4.3 Syringe and Syringe Valves

4.3.1 25-mL glass hypodermic syringes with Luer-Lok (or equivalent) tip (other sizes are acceptable depending on sample volume used).

4.3.2 25-µL micro syringe with a 2 inch x 0.006 inch ID, 22° bevel needle (Hamilton #702N or equivalent).

4.3.3 Micro syringes - 10-, 100-µL.

4.3.4 Syringes - 0.5-, 1.0-, and 5-mL, gas-tight.

4.4 Miscellaneous

4.4.1 Glass vials

4.4.1.1 60-mL, septum-sealed, to collect samples for screening, dry weight determination.

4.4.1.2 40-mL, screw-cap, PTFE lined, septum-sealed. Examine each vial prior to use to ensure that the vial has a flat, uniform sealing surface.

4.4.2 Top-loading balance - Capable of accurately weighing to 0.1 g.

4.4.3 Glass scintillation vials - 20-mL, with screw-caps and PTFE liners, or glass culture tubes with screw-caps and PTFE liners, for dilution of oily waste samples.

4.4.4 Volumetric flasks - Class A, 10-mL and 100-mL, with ground glass stoppers.

4.4.5 2-mL glass vials, for GC autosampler - Used for oily waste samples extracted with methanol or PEG.

4.4.6 Spatula, stainless steel - narrow enough to fit into a sample vial.

4.4.7 Disposable Pasteur pipettes.

4.4.8 Magnetic stirring bars - PTFE- or glass-coated, of the appropriate size to fit the sample vials. Consult manufacturer's recommendation for specific stirring bars. Stirring bars may be reused, provided that they are thoroughly cleaned between uses. Consult the manufacturers of the purging device and the stirring bars for suggested cleaning procedures.

4.5 Field Sampling Equipment

4.5.1 EnCore™ sampler - (En Chem, Inc., 1795 Industrial Drive, Green Bay, WI 54302), or equivalent.

4.5.2 Alternatively, disposable plastic syringes with a barrel smaller than the neck of the soil vial may be used to collect the sample. The syringe end of the barrel is cut off prior to sampling. One syringe is needed for each sample aliquot to be collected.

4.5.3 Portable balance - For field use, capable of weighing to 0.01 g.

4.5.4 Balance weights - Balances employed in the field should be checked against an appropriate reference weight at least once daily, prior to weighing any samples, or as described in the sampling plan. The specific weights used will depend on the total weight of the sample container, sample, stirring bar, laboratory reagent grade water added, cap, and septum.

5.0 REAGENTS

5.1 Organic-free laboratory reagent grade water - All references to water in this method refer to organic-free laboratory reagent grade water.

5.2 Methanol, CH_3OH - purge-and-trap quality or equivalent. Store away from other solvents.

5.3 Sodium bisulfate, $NaHSO_4$ - ACS reagent grade or equivalent.

5.4 Polyethylene glycol (PEG), $H(OCH_2CH_2)_nOH$ – free of interferences at the detection limit of the target analytes.

5.5 See the determinative method for guidance on internal standards and surrogates to be employed in this procedure.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

The low concentration portion of this method employs sample vials that are filled and weighed in the field and never opened during the analytical process. As a result, sampling personnel should be equipped with a portable balance capable of weighing to 0.01 g.

6.1 Preparation of sample vials

The specific preparation procedures for sample vials depend on the expected concentration range of the sample, with separate preparation procedures for low concentration soil samples and high concentration soil and solid waste samples. Sample vials should be prepared in a fixed laboratory or other controlled environment, sealed, and shipped to the field location. Gloves should be worn during the preparation steps.

Because volatile organics will partition into the headspace of the vial from the aqueous solution and will be lost when the vial is opened, surrogates, matrix spikes, and internal standards should only be added to the vials back in the laboratory, either manually by puncturing the septum with a small-gauge needle or automatically by the sample introduction system, just prior to analysis.

6.1.1 Low concentration soil samples

Sodium bisulfate preservation is used in the preparation of vials used in the collection of low concentration soil samples to be analyzed by the closed-system purge-and-trap equipment described in Method 5035.
Water and subsequent freezing preparation of vials is used in the collection of low concentration soil samples known to contain carbonate minerals which may effervesce upon contact with an acidic preservation solution and which are to be analyzed by the closed-system purge-and-trap equipment described in Method 5035. This type of preservation is typically done in the lab after Encore samplers are received from the field

6.1.1.1 Add a clean magnetic stirring bar to each clean vial.

6.1.1.2 The preservative is added to each vial prior to shipping the vial to the field. Add 20 mL of 20% sodium bisulfate solution or 20 mL of water to the vial and seal the vial with the screw-cap and septum seal.

6.1.1.3 Affix a label to each vial. This eliminates the need to label the vials in the field and assures that the tare weight of the vial includes the label. (The weight of any markings added to the label in the field is negligible). It is important that labels and tape not cover the junction of the screw top and vial. Labels and tape must also be applied smoothly (i.e. no wrinkles) to prevent autosampler failures.

6.1.1.4 Weigh the prepared vial to the nearest 0.1 g and record it on the label.

6.1.2 High concentration soil samples in methanol:

6.1.2.1 When high concentration samples are collected without a preservative, a variety of sample containers may be employed, including 40-mL glass vials with septum seals (see Sec. 4.4).

6.1.2.2 The following steps apply to the preparation of vials used in the collection of high concentration soil samples to be preserved in the field with methanol and analyzed by the aqueous purge-and-trap equipment described in Method 5030.

6.1.2.3 Add 20 mL of methanol to each vial.

6.1.2.4 Seal the vial with the screw-cap and septum seal.

6.1.2.5 Affix a label to each vial. This eliminates the need to label the vials in the field and assures that the tare weight of the vial includes the label. (The weight of any markings added to the label in the field is negligible).

6.1.2.6 Weigh the prepared vial to the nearest 0.01 g and record it on the label.

NOTE: Vials containing methanol should be weighed a second time on the day that they are to be used. Vials found to have lost methanol (reduction in weight of >0.01 g) should not be used for sample collection.

6.1.3 Oily waste samples

When oily waste samples are known to be soluble in methanol, sample vials may be prepared as described in Sec. 6.1.2.2, using the appropriate solvent. However, when the solubility of the waste is unknown, the sample should be collected without the use of a preservative, in a vial such as that described in Sec. 6.1.2.1.

6.2 Sample collection

Collect the sample according to the procedures outlined in the sampling plan. As with any sampling procedure for volatiles, care must be taken to minimize the disturbance of the sample in order to minimize the loss of the volatile components. Several techniques may be used to transfer a sample to the relatively narrow opening of the low concentration soil vial. These include devices such as the

EnCore™ sampler, the Purge-and-Trap Soil Sampler™ , and a cut plastic syringe. Always wear gloves whenever handling the tared sample vials.

6.3 Sample handling and shipment

All samples for volatiles analysis should be cooled to approximately 4°C, packed in appropriate containers, and shipped to the laboratory on ice, as described in the sampling plan. Samples should be shipped on the day of sampling if at all possible.

6.4 Sample storage

6.4.1 Once in the laboratory, store samples at 4°C until analysis. The sample storage area should be free of organic solvent vapors.

6.4.2 All samples should be analyzed as soon as practical, and within the designated holding time from collection. Samples not analyzed within the designated holding time must be noted and the data are considered minimum values.

6.4.3 When the low concentration samples are strongly alkaline or highly calcareous in nature, the sodium bisulfate preservative solution may not be strong enough to reduce the pH of the soil/water solution to below 2. Therefore, when low concentration soils to be sampled are known or suspected to be strongly alkaline or highly calcareous, additional steps may be required to preserve the samples. Such steps include: addition of larger amounts of the sodium bisulfate preservative to non-calcareous samples, or the addition of 20 mL of water and storage at -10° (taking care not to fill the vials so full that the expansion of the water in the vial breaks the vial), or significantly reducing the maximum holding time for low concentration soil samples.

7.0 PROCEDURES

This section describes procedures for the low concentration soil method, the high concentration soil method, and the procedure for oily waste samples. High concentration samples are to be introduced into the GC system using Method 5030. Oily waste samples are to be introduced into the GC system using Method 5030 if they are soluble in a water-miscible solvent, or using Method 3585 if they are not.

For the high concentration soil and oily waste samples, the surrogate compounds may either be spiked into the solvent at the time of extraction or the laboratory reagent grade water containing an aliquot of the extract prior to analysis.

7.1 Low concentration soil method (Approximate concentration range of 5 to 200 µg/kg -the concentration range is dependent upon the determinative method and the sensitivity of each analyte.)

7.1.1 Archon Operation

Prior to using this introduction technique for any GC or GC/MS method, the system must be calibrated by the analytical method to be used. When a GC/MS method is used, internal standard calibration is employed.

7.1.1.1 Establish the purge-and-trap instrument operating conditions. Adjust the instrument to inject 10 mL of water, to heat the sample to 40°C, and to hold the sample at 40°C for 1.5 minutes before commencing the purge process, or as recommended by the instrument manufacturer.

7.1.1.2 Carry out the purge-and-trap procedure as outlined in Secs. 7.1.2. to 7.1.4.

7.1.2 Sample purge-and-trap

This method is designed for a 5-g sample size, but smaller sample sizes may be used. The soil vial is hermetically sealed at the sampling site, and MUST remain in order to guarantee the integrity of the sample. Gloves must be worn when handling the sample vial since the vial has been tared. If any soil is noted on the vial or cap, it must be carefully removed prior to weighing. Weigh the vial and contents to the nearest 0.01 g, even if the sample weight was determined in the field, and record this weight. This second weighing provides a check on the field sampling procedures and provides additional assurance that the reported sample weight is accurate. Data users should be advised on significant discrepancies between the field and laboratory weights.

7.1.2.1 Remove the sample vial from storage and allow it to warm to room temperature. Shake the vial gently, to ensure that the contents move freely and that stirring will be effective. Place the sample vial in the instrument carousel according to the manufacturer's instructions.

7.1.2.2 Without disturbing the hermetic seal on the sample vial, add 10 mL of organic-free laboratory reagent grade water, the internal standards, and the surrogate compounds. This is carried out either manually or using the automated sampler. Other volumes of organic-free laboratory reagent grade water may be used. However, it is imperative that all samples, blanks, and calibration standards have exactly the same final volume of organic-free laboratory reagent grade water. Prior to purging, heat the sample vial to 40°C for 1.5 minutes, or as described by the manufacturer.

7.1.2.3 For the sample selected for matrix spiking, add the matrix spiking solution described in Sec. 5.0 of Method 5000, either manually, or automatically, following the manufacturer's instructions.

7.1.2.4 Purge the sample with helium or another inert gas at a flow rate of up to 40 mL/minute (the flow rate may vary from 20 to 40 mL/min, depending on the target analyte group) for 11 minutes while the sample is being agitated with the magnetic stirring bar or other mechanical means. The purged analytes are allowed to flow out of the vial through a transfer line to a trap packed with suitable sorbent materials.

7.1.3 Sample Desorption

7.1.3.1 Non-cryogenic interface - After the 11 minute purge, place the purge-and-trap system in the desorb mode and preheat the trap to 245°C without a flow of desorption gas. Start the flow of desorption gas at 10 mL/minute for about four minutes. Begin the temperature program of the gas chromatograph and start data acquisition.

7.1.4 Trap Reconditioning

After desorbing the sample for 4 minutes, recondition the trap by returning the purge-and-trap system to the purge mode. Maintain the trap temperature at 245°C (or other temperature recommended by the manufacturer of the trap packing materials). After approximately 10 minutes, turn off the trap heater and halt the purge flow through the trap. When the trap is cool, the next sample can be analyzed.

7.2 High concentration method for soil samples with concentrations generally greater than 200 µg/kg.

The high concentration method for soil is based on a solvent extraction. A solid sample is either extracted or diluted, depending on sample solubility in a water-miscible solvent. An aliquot of the extract is added to organic-free laboratory reagent grade water containing surrogates, internal and matrix spiking standards (added manually or by the autosampler), purged according to Method 5030, and analyzed by an appropriate determinative method. The specific sample preparation steps depend on whether or not the sample was preserved in the field. Samples that were not preserved in the field are prepared using the steps below, beginning at Sec. 7.2.1. If solvent preservation was employed in the field, then the preparation begins with Sec. 7.2.4.

7.2.1 When the high concentration sample is not preserved in the field, the sample consists of the entire contents of the sample container. Do not discard any supernatant liquids. Remove a representative aliquot with a spatula.

7.2.2 For soil and solid waste samples that are soluble in methanol, add 5.0 g (wet weight) of sample to a tared 40-mL VOA vial using a calibrated (refer to Katahdin SOP, CA-102, Balance Calibration) top loading balance. Record the weight to 0.1 g. Add 20 mL of methanol to the vial containing the sample and shake for two minutes.

NOTE: The steps in Secs. 7.2.1, 7.2.2, and 7.2.3 must be performed rapidly and without interruption to avoid loss of volatile organics. These steps must be performed in a laboratory free from solvent fumes.

7.2.4 For soil and solid waste samples that were collected in methanol or PEG, weigh the vial to 0.01 g as a check on the weight recorded in the field.

7.2.5 For each new lot of methanol, add an appropriate aliquot of the methanol to 20 mL of organic-free laboratory reagent grade water and analyze by Method 5030 in conjunction with the appropriate determinative method. Proceed to Sec. 7.0 in Method 5030 and follow the procedure for purging high concentration samples.

7.3 High concentration method for oily waste samples

This procedure for the analysis of oily waste samples involves the dilution of the sample in methanol or PEG. However, care must be taken to avoid introducing any of the floating oil layer into the instrument. A portion of the diluted sample is then added to 5.0 mL of organic-free laboratory reagent grade water, purged according to Method 5030, and analyzed using an appropriate determinative method.

The specific sample preparation steps depend on whether or not the sample was preserved in the field. Samples that were not preserved in the field are prepared using the steps below, beginning at Sec. 7.3.2. If methanol preservation was employed in the field, then the preparation begins with Sec. 7.3.4.

7.3.1 For oily samples that are not soluble in methanol or PEG (including those samples consisting primarily of petroleum or coking waste), dilute or extract with hexadecane using the procedures in Method 3585.

7.3.2 For oily samples that are soluble in methanol if the waste was not preserved in the field, tare a 10-mL volumetric flask, or a VOA vial, weigh 1 g (wet weight) of the sample into the tared vessel and add 10.0 mL methanol or PEG with a calibrated syringe. If a vial is used instead of a volumetric flask, it must be calibrated prior to use. This operation must be performed prior to opening the sample vial and weighing out the aliquot for analysis. Invert the vial a minimum of three times to mix the contents.

7.3.4 If the sample was collected in the field in a vial containing methanol or PEG, weigh the vial to 0.1 g as a check on the weight recorded in the field, and proceed with Sec. 7.3.5.

7.3.5 Regardless of how the sample was collected, the target analytes are extracted into the solvent along with the majority of the oily waste (i.e., some of the oil may still be floating on the surface). If oil is floating on the surface, transfer 1 to 2 mL of the extract to a clean GC vial using a Pasteur pipet. Ensure that no oil is transferred to the vial.

7.3.6 Add an appropriate aliquot of the methanol or PEG to 5.0 mL of organic-free laboratory reagent grade water and analyze by Method 5030 in conjunction with the appropriate determinative method. Proceed to Sec. 7.0 in Method 5030 and follow the procedure for purging oily waste samples.

7.4 Determination of % Dry Weight

If results are to be reported on a dry weight basis, it is necessary to determine the dry weight of the sample. Refer to Katahdin SOP, CA-717, for determination of % dry weight.

NOTE: It is highly recommended that the dry weight determination only be made after the analyst has determined that no sample aliquots will be taken from the 60-mL vial for high concentration analysis. This is to minimize loss of volatiles and to avoid sample contamination from the laboratory atmosphere. There is no holding time

associated with the dry weight determination. Thus, this determination can be made any time prior to reporting the sample results, as long as the vial containing the additional sample has remained sealed and properly stored.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

8.1 Before processing any samples, the analyst should demonstrate through the analysis of an organic-free laboratory reagent grade water method blank that all glassware and reagents are interference free. Each time a set of samples is extracted, or there is a change in reagents, a method blank should be processed as a safeguard against chronic laboratory contamination. The blank samples should be carried through all stages of the sample preparation and measurement.

8.2 Initial Demonstration of Proficiency - Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory must also repeat this demonstration whenever new staff are trained or significant changes in instrumentation are made.

8.3 Sample Quality Control for Preparation and Analysis - See the appropriate analytical method to follow to demonstrate acceptable continuing performance on each set of samples to be analyzed. These include the method blank, either a matrix spike/matrix spike duplicate or a matrix spike and duplicate sample analysis, a laboratory control sample (LCS), and the addition of surrogates to each sample and QC sample.

9.0 METHOD PERFORMANCE

Refer to appropriate analytical method.

10.0 APPLICABLE DOCUMENTS/REFERENCES

"Test Methods for the Evaluation of Solid Waste: Physical/Chemical Methods," Method 5035, SW-846, USEPA, Revision III, June, 1997.

TITLE: **CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035**

"Test Methods for the Evaluation of Solid Waste: Physical/Chemical Methods," Method 5035A, SW-846, USEPA, Revision III, June, 1997.

Archon Operator's Manual

LIST OF TABLES AND FIGURES

TITLE: **CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035**

TABLE 1

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-214-04	METHOD 5035, current revision
Apparatus/Materials		
Reagents		
Sample preservation/ handling		
Procedures	(1) Use methanol prep for all high concentration soils. (2) For high concentration soils, leave all extract in the vial with the soil for storage.	(1) For high concentration soils from an unknown source, perform a solubility test. (2) For high concentration soils, pipet approximately 1 mL of extract into a GC vial for storage.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D

Prepared By: Semivolatile Group Date: 02.11.09

Approved By:

Department Manager: [signature] Date: 2-11-09

Operations Manager: Deborah J. Kadeau Date: 2.11.09

QA Officer: Leslie Dimond Date: 02.10.09

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Updated to reflect DoD QSM version 4.1 compliance and new standard preparation procedures.	EH	08/09	08/09

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document SOP CA-226-01, titled "Analysis of Semivolatile Organic Compounds by Capillary Column GC/MS: SW 846 Method 8270D".

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document SOP CA-226-01, titled "Analysis of Semivolatile Organic Compounds by Capillary Column GC/MS: SW 846 Method 8270D".

Recipient: _____ Date: _____

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures utilized by Katahdin Analytical Services, Inc. laboratory personnel to prepare and analyze water and soil sample extracts for semivolatile organics by EPA SW-846 Method 8270D.

In order to maintain consistency in data quality, this SOP consolidates all aspects of the analyses in one working document, to be revised as necessary.

1.1 Definitions:

ANALYTICAL BATCH: 20 or fewer samples which are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods.

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, laboratory reagent grade water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

CALIBRATION CHECK: Verification of the ratio of instrument response to analyte amount; a calibration check is done by analyzing for analyte standards in an appropriate solvent. Calibration check solutions are made from a stock solution, which is different from the stock used to prepare standards.

INDEPENDANT CALIBRATION STANDARD: A solution prepared from a stock standard solution independent of the standard that is used to calibrate the instrument. Analyzed immediately after calibration,

CALIBRATION STANDARD (WORKING STANDARD): A solution prepared from the stock standard solution, which is used to calibrate the instrument response with respect to analyte concentration.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed.

Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

STANDARD CURVE (CALIBRATION CURVE): A curve that plots concentration of known analyte standard versus the instrument response to the analyte.

STOCK STANDARD SOLUTION: A concentrated solution containing a single certified standard that is a method analyte, or a concentrated solution of a single analyte prepared in the laboratory with an assay reference compound. Stock standard solutions are used to prepare calibration standards.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

TARGET: A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

TARGET DB: An oracle database used to store and organize all Target data files.

QUICKFORMS: A laboratory reporting software for Target and Target DB. The QuickForms report module for Target is preconfigured with generalized forms and US EPA CLP report forms and disk deliverables, which can be customized.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of semivolatile organic compounds by EPA Method 8270D. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, “Personnel Training & Documentation of Capability,” current revision.

It is the responsibility of all Katahdin technical personnel involved in analysis of semivolatiles by Method 8270D to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

After analysis, autosampler vials containing sample extracts in methylene chloride are returned to the SVOA hood, and the contents transferred to a labeled waste container. The contents of this container are disposed of in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

2.0 SUMMARY OF METHOD

The process involves the extraction of semivolatiles from a sample using an appropriate solvent followed by clean up steps (where applicable) and concentration of the extract (refer to Katahdin SOP CA-502, “Preparation Of Aqueous Samples For Extractable Semivolatile Analysis”, SOP CA-512, “Preparation Of Sediment/Soil Samples By Sonication Using Method 3550 For Subsequent Extractable Semi-Volatiles Analysis” and SOP CA-526, “Preparation Of Sediment/Soil Samples By Soxhlet Extraction Using Method 3540 For Subsequent Extractable Semivolatile Analysis”). An aliquot of the final extract is injected into the gas chromatograph for compound separation by capillary column, followed by the

electron impact mass spectrometer for identification and quantitation.

Target and surrogate compounds are identified and compared to the mass spectra obtained from the analysis of standard solutions containing the same compounds. A relative response factor is established for each target compound and surrogate against an internal standard during the most recent initial or continuing calibrations. The identified compound is then quantitated using the relative response factor, the amount of internal standard in the sample, the initial volume of sample, and any other factors, such as dilutions.

3.0 INTERFERENCES

Interfering contamination may occur when a sample containing low concentrations of SVOCs is analyzed immediately after a sample containing high concentrations of SVOCs. Any samples that have suspected carryover must be reanalyzed.

4.0 APPARATUS AND MATERIALS

4.1 GC: Hewlett Packard 5890 and/or 6890

4.2 Mass Spectrometers (MS): HP5973, HP5972 and/or HP5970

4.3 Helium: Carrier gas for routine applications. All carrier gas lines must be constructed from stainless steel or copper tubing; non-polytetrafluoroethylene (non-PTFE) thread sealant or flow controllers with rubber component are not to be used.

4.4 Autosamplers: HP 7673As and HP 7683s

4.5 Hamilton syringes: 2.00 uL to 10 mL

4.6 Volumetric glassware: Grade A or equivalent

4.7 Columns: DB-5MS 30m, 0.25mm I.D., 25um film thickness, columns (J&W Scientific) or equivalent.

4.8 Acquisition System: The acquisition system must be interfaced to the MS and allow continuous acquisition of data throughout the duration of the chromatographic program. It must permit, at a minimum, the output of time vs. intensity (peak height or peak area). Hewlett Packard Chemstation or equivalent.

4.9 Data System: The Target software is used for processing data and generating forms.

4.10 1.8 mL vials with 350uL inserts

4.11 Crimp tops with Teflon lined septa

5.0 REAGENTS

5.1 J.T. Baker Ultra Resi-Analyzed methylene chloride (or equivalent)

5.2 Purge and trap grade methanol

5.3 Standards: Stock standards and working standards are received and recorded in accordance with SOP CA-106 “Standard Preparation and Documentation”.

5.3.1 The expiration date for all standards is one year from date of opening the ampule. If the manufacturer’s expiration date is before this one year date, the manufacturer’s expiration must be followed. New standards must be opened if degradation is observed.

5.3.2 Secondary dilution standards

The standards are prepared on an as needed basis (but not less than every 6 months) and stored in screw cap amber bottles with Teflon liners in the BNA standards freezer between uses. Standards prepared from various stock solutions must always use the first expiration date of any of the solutions used for preparation.

5.3.2.1 Calibration Mix – Prepare a standard stock mix that contains those compounds commonly considered 8270 and those compounds commonly considered Appendix IX compounds. The compound dinoseb should <u>not</u> be added to this stock as it is only available in methanol. This will be added separately to each calibration level. Use Table 3 as a guide. The stock should be prepared at 125 ug/mL.

5.3.2.2 Independent Calibration Verification (ICV) Standard – From a source other than that used to make the calibration standards, prepare separate standards mixes (A and B) such that Standard Mix A contains those compounds commonly considered 8270 and Standard B Mix contains those compounds commonly considered Appendix IX compounds. Use Table 3 as a guide. Each stock should be prepared at 100 ug/mL.

5.3.2.3 DFTPP Solution – Prepare standard in methylene chloride containing DFTPP, Pentachlorophenol, Benzidine and DDT at a final concentration of 25 ug/mL.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

All semivolatile sample extracts should be refrigerated until analysis. Extracts must be analyzed within forty days following the date of extraction.

7.0 PROCEDURES

7.1 NAMING AND CODING CONVENTIONS FOR ANALYTICAL STANDARDS – Used in accordance with SOP CA-106 “Standard Preparation and Documentation”.

7.2 COMPUTER (DATA SYSTEM) CONVENTIONS -

Conventions for all instruments are as follows:

Sub-Directory for data acquisition and storage: C:\HPCHEM\1\DATA

Tune file: DFTPP.U

Method files: L8270CXX.M (all samples and standards)

Where:
XX = the calibration number in chronological order
L = instrument ID (R, U, or G)

DFTPP tuning acquisition: DFTPP390.M

NOTE: All acquisition parameters must be identical for L8270CXX.M and DFTPP390. M.

Data Files: L_ _ _ _.D, where _ _ _ _ is a number in chronological order from 0001 to 9999 and L is the instrument ID (R, U, or G). This file also contains the Quantitation output file.

Data Files for DFTPP: LD_ _ _.D, where _ _ _ is a number in chronological order from 001 to 999 and L is the instrument ID (R, U, or G).

7.3 INSTRUMENT SPECIFIC PROCEDURES

It is the policy of the GC/MS group that all data be acquired in the batch mode. The following items must be checked prior to data acquisition in the batch mode:

- Ensure that the proper sequence and tune files are being used.
- Check the autosampler syringe (Is it clean? Does the plunger move freely? etc.), its alignment and make sure the solvent rinse vial is full. Ensure that the knurled nut holding the top of the syringe plunger is tight.

- Look at the batch to be analyzed and check the following:
  - Make sure that the data files are in numerical order with no duplication and that the method file is the same as that used for ICAL or Continuing Calibration analysis.
  - Bottle numbers match with the numbers on the autosampler tray.

After the batch has been deemed free of errors, start the batch by using the "Position and run" command under the SEQUENCE menu in MSTop.

7.4 INSTRUMENT TUNING - Prior to the analysis of any calibration standards, blanks or samples, the GC/MS system must be shown to meet the mass spectral key ion and ion abundance criteria for decafluorotriphenylphosphine (DFTPP) tabulated below. Pentachlorophenol, benzidine and DDT are also present in this standard.

Mass	Criteria
51	30 to 60% of mass 198
68	< 2% of mass 69
70	< 2% of mass 69
127	40-60% of mass 198
197	< 1% of mass 198
198	base peak, 100 % relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	> 1% of mass 198
441	present but less than mass 443
442	> 40% of mass 198
443	17-23% of mass 442

All ion abundances must be normalized to m/z 198, the nominal base peak.

The following are the GC/MS operating conditions for injection of DFTPP.

Initial column temperature hold	140°C for 3 minutes
Column temperature program	140-275°C at 15 degrees/minute
Final column temperature hold	275°C
Injection port temperature	280°C
Transfer line/source temperature	285°C
Injector - splitless, valve time	0.18 minutes
EPC	inlet B
Constant flow	ON
Constant flow pressure	10psi
Constant flow temperature	30°C
Vacuum comp.	ON

Run time	10-12 minutes
Scan start time	5.0-6.0 minutes
Sample volume	2.0 uL of 25 ng/uL DFTPP solution
Carrier gas	helium at approximately 60 mL/minute
Mass range	35 to 500 amu
Number of A/D samples	4
GC Peak threshold	500 counts
Threshold	10 counts

Set up the run on the Enviroquant system using "Edit Sample Log Table". For a more detailed explanation of the Enviroquant software, consult the appropriate manual, Department Manager, or senior chemist within the GC/MS group.

When the DFTPP has concluded, the run must be evaluated to determine if sample analysis can proceed. The chromatography and the ion ratios must be examined. The DFTPP run is processed using the current algorithms in the Target software.

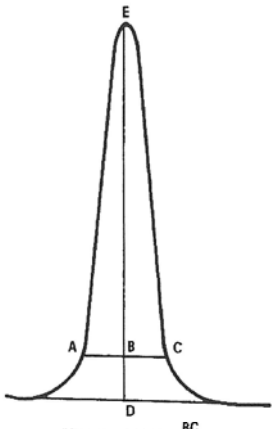
If the results indicate the system does not meet acceptance criteria, the GC/MS must be manually tuned. Once the manual tune procedure is completed, DFTPP must be re-injected and reevaluated. If the instrument still does not meet criteria, notify your Department Manager. Under no circumstances should calibration proceed if the instrument DFTPP is not in criteria.

The DFTPP tuning standard should also be used to assess the column performance and injection port inertness. Calculate the degradation of DDT to DDE and DDD; it should not exceed 20%. Benzidine and pentachlorophenol should be present at their normal responses, and should not exceed a tailing factor of 2 given by the followint equation:

$$\text{Tailing Factor} = \frac{BC}{AC}$$

.Where: AC = the width at 10% height
DE = height of the peak
B = the height at 10% of DE

Example:



In order to document the performance of benzidine, pentachlorophenol and DDT, the following procedure must be followed. At the PC, which operates the instrument, load the method TUNETAIL.M into the ENVDA screen. Go into the quant drop down menu and select *calculate/generate report*. When that finishes, select *Qedit quant result.* Each compound can now be evaluated. Double click on benzidine and select *ChromEval* and then *Evaluate tailing*. Follow the instructions given on the screen to evaluate tailing. Send the report to the printer. Repeat the procedure for pentachlorophenol. Repeat the procedure for DDT, selecting *Evaluate degradation*. Follow the instructions given on the screen and then send the report to the printer. The report should be filed with the tune raw data.

The DFTPP solution must be analyzed once at the beginning of each twelve hour period during which standards and/or samples are analyzed. The 12 hour time period for GC/MS system begins at the moment of injection of the DFTPP analysis. The time period ends after twelve hours has elapsed according to the system clock. The last injection must be accomplished prior to the expiration of 12 hours; conceivably, the run-time of an injection could end after the twelve hours.

7.5 INSTRUMENT CALIBRATION

7.5.1 Initial Calibration for Method 8270D

Prior to the analysis of samples and required method blanks, and after the instrument DFTPP tuning criteria have been met, the GC/MS system must be calibrated. The calibration consists of a six point curve. The calibration

levels are 10, 25, 50, 75, 100 and 125 ng/uL Calibration is done to determine instrument sensitivity and the linearity of GC/MS response for the semivolatile target and surrogate compounds.

Final conc. (ng/uL)	125ng /uL SVOA Stock Soln Added (uL)	1000 ug/mL dinoseb Standard (uL)	$MeCl_2$ Added (uL)	Final Vol (uL)	IS Added (uL)
10	16	2	182	200	2
25	40	5	155	200	2
50	80	10	110	200	2
75	120	15	65	200	2
100	160	20	20	200	2
125	100	0	0	100	1

If additional compound mixtures are added, the volume of $MeCl_2$ is adjusted to maintain a final volume of 200 or 100 uL. A 100 uL aliquot of each of the standards above is spiked as above with 4000 ng/uL Internal Standard stock and analyzed.

Internal Standards
1,4-Dichlorobenzene-d4
Naphthalene-d8
Acenaphthene-d10
Phenanthrene-d10
Chrysene-d12
Perylene-d12

The GC/MS operating conditions for the calibration standards injections are the same as for the DFTPP with the following exceptions:

Column Temperature Program	40°C for 3 minutes to 300°C at 10°/minute
Final Column Temperature hold	300°C
Run Time	34-36 minutes
Scan Start Time	1.8 minutes – variable, depending upon column length
Injection volume	1 uL

The conditions are set up in the method files L8270CXX.M.
After analysis of the six calibration points, they must be processed and evaluated for adherence to QC criteria. Minimum requirements of ID files are the use of specific quantitation ions and quantitating a specific set of targets and surrogates with a set internal standard. These requirements are found in Tables 3 and 5.

7.5.2 Initial Calibration Criteria

Relative response factors (RRFs) must be calculated and evaluated for each target compound and surrogate. The RRF is defined as follows:

$$RRF = \frac{Ax}{A_{IS}} \times \frac{C_{IS}}{Cx}$$

where: Ax = area of the primary ion for the target compound
A_{IS} = area of the primary ion for the corresponding istd
C_{IS} = concentration of the istd (ng/uL)
C_x = concentration of the target compound

After the calibration points have been quantitated, update the calibration curve points using the Target data processing software to generate the Mean RRF and %RSD for all analytes. If information is needed concerning the use of these programs, consult the Department Manager or a senior chemist within the group.

Response factor criteria have been established for the calibration of the semivolatile target and surrogate compounds. These criteria must be met in order for the calibration curve to be considered valid. The percent RSD for each target analyte must be less than or equal to 20%.

It is recommended that a minimum response factor (Table 6) for target analytes be achieved as a means to ensure that these compounds are behaving as expected. In addition, meeting the minimum response factor for the lowest calibration standard is critical in establishing and demonstrating the desired sensitivity. Therefore the minimum response factors in Table 6 must be verified at the lowest calibration level.

7.5.2.1 Linearity of Target Analytes (This is also applicable to clients that request DOD criteria.)

If the RSD of any target analyte is 20% or less, then the response factor is presumed to be constant over the calibration range, and the average response factor may be used for quantitation.

If the RSD of any target analyte exceeds 20%, then a calibration option outlined in section 7.0 of method 8000 will need to be employed. Please note that some options may not be allowable for certain states, federal programs, or clients. South Carolina does not allow option 2 (non-linear) for compliance work originating in their state.

Option 1 (Section 7.5.2 of method 8000 - Rev. 2, 12/96), is a linear

regression of instrument response versus the standard concentration. The correlation coefficient (r) for each target analyte and surrogate must be greater than or equal to 0.995. For linear models, Target reports r^2. This is calculated by either calculating r or squaring the result or by calculating the coefficient of determination. For a linear calibration, the equation for either is the same. The value for r^2 must greater than or equal to 0.990.

The method of linear regression analysis has the potential for a significant bias to the lower portion of a calibration curve. When calculating the calibration curves using the linear regression model, a minimum quantitation check on the viability of the lowest calibration point should be performed by re-fitting the response from the low concentration calibration standard back into the curve. The recalculated concentration of the low calibration point should be within ± 30% of the standard's true concentration. Analytes which do not meet the minimum quantitation calibration re-fitting criteria should be considered "out of control".

Corrective action such as redefining the lower limit of quantitation and/or reporting those "out of control" target analytes as estimated when the concentration is at or near the lowest calibration point may be appropriate.

Option 2 (Section 7.5.3 of method 8000 - Rev. 2, 12/96), is a non-linear calibration model not to exceed a third order (seven calibration points required) polynomial. The lab would use a quadratic model or second order polynomial. The use of a quadratic model requires six calibration points. In order for the quadratic model to be acceptable, the coefficient of determination must be greater than or equal to 0.99.

If more than 10% of the compounds in the initial calibration exceed the 20% RSD limits and do not meet the minimum correlation coefficient of determination criteria in option 1 or 2, the GCMS system is considered out of control and the calibration must be repeated. Note: Maintenance may have to be performed.

Internal standard (IS) responses and retention times in all standards must be evaluated immediately after data acquisition; if the RT for any IS changes by more than 0.50 minutes from the latest daily calibration standard, corrections must be made to the chromatographic system. If the extracted ion current profile (EICP) area for any IS changes by more than a factor of two (-50% to +100%), corrective action must be performed.

Each GC/MS system must be calibrated following system corrective

action, including ion source cleaning or repair and column removal or replacement.

If time remains in the clock after meeting the initial calibration acceptance criteria, samples may be analyzed. The calibration must be verified each twelve hour time period (time period starts from the moment of the DFTPP injection) for Method 8270. The SSTD050 in the curve may be used as the calibration verification standard as long as it meets the calibration verification acceptance criteria. All sample results must be quantitated using the initial calibration response factors.

7.5.2.2 Immediately following calibration an Independent Calibration Verification Standard must be analyzed. The percent difference for each target analyte must be less than or equal to 30%. For clients requiring DOD criteria, all project analytes must be within +/- 25% of true value.

7.5.2.3 Retention Time Windows

Retention time windows are set at the midpoint standard of the calibration curve, following every ICAL. When a CV is analyzed (and not an ICAL), the retention time windows of the daily CV must be within 30 seconds of the midpoint calibration standard of the most recent ICAL. The samples analyzed following the daily CV must have retention times within 30 seconds of those for the daily CV. Each successive daily CV must be compared to the most recent ICAL midpoint standard.

7.5.3 Continuing Calibration

A calibration verification check standard must be performed once every twelve hours immediately following analysis of the tuning compound DFTPP. This check contains all target compounds and surrogates at a concentration of 50 ng/uL.

After quantitation of the 50 ng/uL continuing calibration check, response factors must be calculated and compared to the average response factors in the initial calibration. The Target program calculates the calibration check response factors and compares them to the average RFs in the calibration curve by calculating percent differences.

- All target analytes must have a % difference of +/- 20%D in order to be considered in criteria.

- All target analytes should meet the minimum RRF criterion as in ICAL

(Table 6) in order to be considered in criteria.

These conditions must be met before method blank and/or sample analysis can begin.

The area for the internal standards in the calibration verification must be within a factor of two (-50% to 100%) from the mid-point standard of the most recent initial calibration. This is listed in the ISTD monitor report.
If the calibration verification does not meet criteria, corrective action must be taken. Depending on the situation, corrective action may be as follows:

- Re-analyze the 50 ng/uL continuing calibration check.
- Change the septum; clean the injection port; install a clean, silanized quartz liner; cut off a small portion (1" to 3") of the front end of the capillary column (this is usually performed when acid RFs are low and/or chromatography is poor).
- Analyze a new initial calibration curve.

The last option, the generation of a new initial calibration curve, is usually chosen when percent difference are >30%. In these instances, there is little or no chance of a continuing calibration reanalysis meeting criteria. If there is any doubt concerning which corrective action to undertake, consult the Department Manager or a senior chemist within the group.

If the calibration verification does meet the criteria specified above then analysis may proceed using initial calibration response factors.

7.6 SAMPLE ANALYSIS

Sample extracts may be analyzed only after the GC/MS system has met tuning criteria, initial calibration and continuing calibration requirements. Ensure that the same instrument conditions are being used for tuning, calibration and sample analysis
by reviewing the GC parameters using the “Edit entire method” option under the Method menu in MSTOP. Note that you can not edit a method if the instrument is running.

Extracts are stored in the refrigerator in the organics extraction laboratory at 4°C ±2°C. Remove them from the refrigerator and place them in the GC/MS laboratory semivolatile hood when ready for analysis.

Prepare a 1.8 mL clear glass vial (crimp top) with a disposable insert (350 uL). Add 100 uL of sample extract and 1.0 uL of the 4000 ng/uL IS stock to the vial and then cap. This gives a 40 ng/uL final concentration for the internal standard compounds.

The samples are topped with Teflon lined crimp top caps.

7.7 FINAL DATA PACKAGE

7.7.1 Initial Data Review (IDR)

The initial data review is accomplished by the analyst who analyzed the samples and is a review of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed. The initial data review is performed on the detailed quantitation reports of the analyzed sample. This data review examines criteria that directly impact whether or not the sample needs to be reanalyzed:

- Surrogate Recoveries
- Internal Standard Area Stability
- Method Blank Acceptance
- Chromatography
- Target Compound Detection/Quantitation/Review for false positives
- Laboratory Control Sample Recoveries
- Matrix Spike/Matrix Spike Duplicate Recoveries

The analyst must evaluate all data using the QA Acceptance Criteria table found within this SOP (Table 1). This table gives acceptance criteria and corrective actions for criteria that are not met. In addition to evaluating QC elements, the chromatography and quantitation of target analytes must be reviewed. During this review, the analyst checks the integration of each individual peak. The hardcopy has false positives crossed out so they can be reviewed for appropriateness by the Department Manager.

7.7.2 Chromatography

The chromatography should be examined for the presence or absence of any ghost peaks and can also be used as an indication of whether or not matrix interferences might be affecting surrogate recoveries and/or Istd area recoveries. Whether or not the chromatography is acceptable is a judgment call on the part of the analyst and should be used in conjunction with other monitored QC (e.g. surrogate recoveries) to determine the necessity of reanalyzing.

Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating the peak of concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.

Each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. Should a manual

integration be necessary (for instance, due to a split peak, peak tailing, or incomplete resolution of isomeric pairs), an "m" qualifier will automatically be printed on the quantitation report summary. All manual integrations are initialed, dated and given a code which describes the reason for the manual integration.

This manual integration package must then be submitted to the Department Manager or his/her designee, who will review each manual integration.
For specific procedures on how to manually integrate, refer to Katahdin SOP QA-812, "Manual Integration", current revision.

7.7.3 Target Compound Detection/Quantitation
The semivolatile ID files have been set up to err on the side of false positives; that is, to identify and quantitate peaks as target compounds that may not necessarily be valid hits. It is the responsibility of the GC/MS analyst to use his/her technical judgment to determine if the identification of a target compound is correct or not.

If any target concentration exceeds the upper limit, a dilution must be made and analyzed. The dilution chosen should keep the concentration of the largest target compound hit in the upper half of the initial calibration range. LCS and MS/MSD samples need not be diluted to get spiked analytes within the calibration range.

The requirements for qualitative verification by comparison of mass spectra are as follows:

- All ions present in the standard mass spectra at a relative intensity > 10% must be present in the sample spectrum.
- The relative intensities of primary and secondary ions must agree within ±20% between the standard and sample spectra.
- Ions greater than 10% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst.

If a compound cannot be verified by all three criteria above, but, in the technical judgment of the mass spectral interpretation specialist, the identification is correct, then the laboratory shall report that compound on the Form 1 as a valid hit.

The GC/MS laboratory initial data review must be completed within twelve hours of batch completion; in the majority of instances, the initial review should be accomplished at the beginning of a work shift for the previous set of analyses.

7.7.3.1 Tentatively Identified Compounds (TIC)

TIC's may be requested by certain clients for samples. Refer current Katahdin to SOP CA-207 "GC/MS Library Search and Quantitation.

7.7.4 Reporting

After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into QuickForms. Depending on the QC label requested by the client, a Report of Analysis (ROA) and additional reports, such as LCS forms and chronology forms, are generated. The package is assembled to include the necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded to the secondary reviewer. The secondary reviewer validates the data and checks the package for any errors. When completed, the package is sent to the department manager for final review. A complete review checklist is provided with each package. The final data package from the Organics department is then processed by the Data Management department.

7.8 Injection Port Liner Cleaning And Silanizing Procedure

7.8.1 Remove the rubber o-ring from the liner and place the liner in a large Erlenmeyer flask.

7.8.2 In the hood, pour nitric acid into the flask until the liner is covered. Place the flask on a hotplate and boil for 2-3 hours.

7.8.3 Let cool; drain nitric acid and thoroughly flush the liner with water.

7.8.4 Bake briefly in the muffle oven until liner is dry and cool to room temperature.

7.8.5 Place the liner in a beaker, fill with Sylon and let it soak for at least two hours.

7.8.6 Take out the liner and rinse it thoroughly with toluene.

7.8.7 Rinse the liner thoroughly with purge and trap grade methanol.

7.8.8 Bake the liner in the muffle oven for a minimum of three hours.

7.9 Instrument Maintenance

Instrument preventative maintenance is performed on a semi-annual basis by GC/MS chemists. This maintenance includes a thorough inspection and cleaning of all parts, including changing rough and turbopump oils. GC/MS analysts perform other maintenance on an as-needed basis. Typically, routine maintenance involves

clipping off the front end of the DB-5MS column, replacing the injection port septum, and installing a freshly silanized quartz liner after sample analysis.

All maintenance must be documented in the instrument-specific maintenance log, whether it is routine or not. The Department Manager must authorize any maintenance over and above a routine source cleaning.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 and to details in this section for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The criteria does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in this section or in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in this section and in Table 1 may rely on analyst experience to make sound scientific judgements. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.
In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 Method Blank Criteria

A method blank is defined as a volume of a clean reference material (laboratory reagent grade water for water samples, baked organic-free sand for soil/sediment matrices) that is carried through the entire analytical procedure. One method blank must be extracted with each group of samples of a similar matrix and must be analyzed on the GC/MS system that was used to analyze the samples.

An acceptable method blank must contain less than or equal to the PQL of any target compound. For clients requiring DOD criteria, no analytes detected at > ½ PQL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit.

If the method blank exceeds these contamination levels, the analytical system is considered out of control and corrective action must be taken before sample analysis.
Reanalysis of the blank is the first step of the corrective action; if that does not solve the problem, a Katahdin Corrective Action Report (CAR) will be initiated.

Corrective action will be specified after consultation including the Department Manager, Operations Manager, and QA Officer.

8.2 Surrogate Recoveries

There are six surrogates, which can be divided as follows:

- B/N - Nitrobenzene-d5, 2-Fluorobiphenyl and Terphenyl-d14
- Acid - Phenol-d5, 2-Fluorophenol and 2,4,6-Tribromophenol

The surrogates have laboratory derived statistical limits that are updated on an annual basis and are available in the QA office. For clients requiring DOD criteria, use acceptance limits specified by DOD or use in-house limits where none are specified.

If specifications are not met, the sample (or blank) should be reanalyzed. If specifications are met in the reanalysis, this reanalysis should only be submitted. If surrogate specifications are not met in the sample or method blank reanalysis, a Corrective Action Report (CAR) should be initiated. Corrective action will be specified after consultation including the Department Manager and Operations Manager.

For further information regarding the acceptance of surrogate recoveries, consult the Department Manager.

8.3 Internal Standard Responses

Internal standard responses and retention times (RT) in all samples and blanks must be evaluated as part of the technical data review. The method files have been set up to only detect compounds that fall within a set RT window. For Method 8270 analysis, if the extracted ion current profile (EICP) area for any internal standard changes by more than a factor of two (-50% to +100%) as compared to the daily continuing calibration standard, reanalysis must occur. If the reanalysis meets criteria, only the in-criteria run should be reported. If the reanalysis is still out-of-criteria, both analyses should be included in the sample package set.

MS/MSD samples that do not meet the EICP area criteria above do not have to be reanalyzed.

8.4 Laboratory Control Sample (LCS)

An LCS must be performed for each group of samples of a similar matrix, for the following, whichever is more frequent:

- Every 20 samples of a similar matrix or similar concentration, or
- Every batch of samples extracted.

Statistical limits are compiled annually for LCS recoveries (archived in QA office). Statistical limits are only calculated when at least 20 usable data points are obtained for any given compound. If insufficient data points are available, nominal limits are set by the Department Manager, Laboratory Operations Manager and Quality Assurance Officer. Refer to Katahdin SOP QA-808, "Generation and Implementation of Statistical QC Limits and/or Control Charts", current revision.

The use of statistical limits versus nominal limits is dependent on the client and project. This information is communicated to the Department Manager through the Katahdin project manager. It is standard practice to use statistical limits for reporting purposes and to evaluate any QC criteria exceedances. However, nominal limits of 60-140% or 70-130% may be used for some projects or states (i.e. South Carolina). For clients requiring DOD criteria, use acceptance limits specified by DOD or use in-house limits where none are specified.

The LCS recoveries for all analytes are evaluated. All of the compounds of interest must fall within the established statistical limits with the following sporadic exceedance allowances. South Carolina does not allow for marginal exceedances for compliance work originating in their state.

Number of Analytes	Number of Allowable Exceedances
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
<11	0

If less than the number of allowable exceedances fail the statistical limits, no corrective action is needed. If greater than the number of allowable exceedances fail the statistical limits, corrective action may be taken. Corrective actions may vary with each situation. However, in the case where the failures are high and the samples are non-detect for those compounds, then no corrective action is required. Otherwise, corrective action may involve reanalysis or recalibration. The specific corrective actions taken will rely on analyst experience to make sound scientific judgments while considering client objectives, other quality control indicators and/or the ability to reanalyze a sample within holding time.

Please note that for compounds with only nominal limits (i.e. insufficient data points

were available to generate statistical limits), no corrective action is required for out-of-criteria recoveries until enough data points are established to generate statistical limits.

8.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Criteria

Matrix Spike and Matrix Spike Duplicates must be extracted and analyzed for each group of up to 20 samples of a similar matrix or similar concentration. In the event insufficient sample volume is available an LCS/LCS Duplicate is extracted and analyzed in place of the MS/MSD.

Statistical limits are compiled annually for MS/MSD recoveries for a short list of the spiked compounds. Nominal limits of 60-140% are used for all other compounds. Generally, corrective action is only taken for the short list of the spiked compounds. The specific corrective actions will rely on analyst experience to make sound scientific judgements while considering client objectives, other quality control indicators and/or the ability to reanalyze a sample within holding time. For clients requiring DOD criteria, use acceptance limits specified by DOD or use in-house limits where none are specified.

8.6 QC Requirements

Refer to Table 1 for a summary of QC requirements, acceptance criteria, and corrective actions. Table 1 criteria are intended to be guidelines for analysts. The table does not over all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all of the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The Department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Samples may not be able to be reanalyzed within hold time. In these cases “qualified” data with narration may be advisable after consultation with the client.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Organic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

Refer to the current revision of Method 8270 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007, Method 8270D.

Katahdin SOP CA-101, Equipment Maintenance, current revision.

Department of Defense Quality Systems Manual for Environmental Laboratories (DoD QSM), Version 4.1, 04/22/09

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003

LIST OF TABLES AND FIGURES

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

TABLE 1

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Check of mass spectral ion intensities using DFTPP	Prior to initial calibration and calibration verification	Refer to the criteria listed in Section 7.4	Retune instrument, and verify
Six-point initial calibration for all analytes	Initial calibration prior to sample analysis	RSD 20% for all compounds. If not met: Option 1) Linear least squares regression: $r \geq 0.995$ Option 2) Non-linear regression: coefficient of determination (COD) $r^2 \geq 0.99$ (6 points for second order) Up to 10% target analytes may be outside of the above criteria Refer to section 7.5.2.1 for additional information.	Perform instrument maintenance if necessary. Repeat calibration if criterion is not met
Independent calibration verification	Once after Initial calibration	± 30 % D	1) Reanalyze standard 2) Reprep standard 3) Reprep standard from fresh stock.
Continuing calibration verification	Once per each 12 hours, prior to sample analysis	All target analytes: $\leq$ 20%D	Repeat initial calibration and reanalyze all samples analyzed since the last successful calibration verification
ISs	Immediately after or during data acquisition of calibration check standard	Retention time $\pm$ 30 seconds; EICP area within -50% to +100% of last calibration verification (12 hours) for each IS	Inspect mass spectrometer or GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning
Demonstration of ability to generate acceptable accuracy and precision	Once per analyst and annually there after.	All recoveries within method QC acceptance limits.	Recalculate results; locate and fix problem; reextract/reanalyze P&A study for those analytes that did not meet criteria
Method blank	One per prep batch	No analytes detected > PQL	1) Investigate source of contamination 2) Evaluate the samples and associated QC: i.e.If the blank results are above the PQL, report samples that are <PQL or > 10X the blank result. Reprep a blank and the remaining samples.

TABLE 1, (cont.)

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
LCS for all analytes	One LCS per prep batch	Statistically derived from lab data or nominal limits depending on the project. Refer to QA records for statistical limits. Nominal limits are used as default limits. See also section 8.4 of this SOP for more information on allowable exceedances.	1) Evaluate the samples and associated QC: i.e.If an MS/MSD was performed and acceptable, narrate. 2) If an LCS/LCSD was performed and only one was unacceptable, narrate. 3) If the surrogate recoveries in the LCS are low but are acceptable in the blank and samples, narrate. 4) If the LCS rec. is high but the sample results are <PQL, narrate. 5) Otherwise, reprep a blank and the remaining samples.
Surrogate spike	Every sample, control, standard, and method blank	Current statistical limits	1) Check chromatogram for interference; if found, flag data 2) If not found, check instrument performance; if problem is found, correct and reanalyze 3) If still out reextract and analyze sample 4) (4) If reanalysis is out, flag data
MS/MSD	One MS/MSD per every 20 samples	Statistically derived from lab data or nominal limits depending on the project. Refer to QA records for statistical limits and section 8.5 of this SOP.	1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. 2) (2) If both the LCS and MS/MSD are unacceptable reprep the samples and QC.
MDL study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

Topic	Katahdin SOP CA-226-01	Method 8270, current revision
Apparatus/Materials	none	
Reagents	none	
Sample preservation/ handling	none	
Procedures	none	
QC - Spikes	none	
QC - LCS	none	
QC - Accuracy/Precision	none	
QC - MDL	PQL – Practical Quantitation Level – three to ten times the MDL.	EQL – Estimated Quantitation Level – five to ten times the MDL

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

TABLE 3

Analyte Quantitation and Internal Standards

Internal Standard: 1,4-dichlorobenzene-d4

Target and Surrogates:

Pyridine (not on TCL list)
N-Nitrosodimethylamine (not on TCL list)
Aniline (not on TCL list)
Phenol
Bis (2-chloroethyl) ether
2-Chlorophenol
1,3-Dichlorobenzene
1,4-Dichlorobenzene
1,2-Dichlorobenzene
Benzyl alcohol (not on PP list)
2-Methylphenol (not on PP list)
2,2'-oxybis(1-chloropropane) (also known as Bis (2-Chloroisopropyl) ether)
4-Methylphenol (not on PP list)
N-Nitroso-di-n-propylamine
Hexachloroethane
Ethyl methanesulfonate (8270 C)
Methyl methanesulfonate (8270 C)
2-Picoline (8270 C)
N-Nitrosomethylethylamine (Appendix IX)
N-Nitrosodiethylamine (Appendix IX)
N-Nitrosopyrrolidine (Appendix IX)
N-Nitrosomorpholine (Appendix IX)
2-Fluorophenol (surrogate)
Phenol-d6 (surrogate)

Internal Standard: Naphthalene-d8

Target and Surrogates:

Nitrobenzene
Isophorone
2-Nitrophenol
2,4-Dimethylphenol
Acetophenone (8270 C)
Benzoic acid (not on PP list)
Bis (2-chloroethoxy) methane
2,4-Dichlorophenol
2,6-Dichlorophenol (8270 C)
1,2,4-Trichlorobenzene
a, a-Dimethyl-phenethylamine (8270 C)
Naphthalene
4-Chloroaniline (not on PP list)
Hexachlorobutadiene
4-Chloro-3-methylphenol
2-Methylnaphthalene
N-Nitrosodi-n-butylamine (8270 C)
N-Nitrosopiperidine (8270 C)
o-toluidine (Appendix IX)
o, o, o-Triethylphosphorothioate (Appendix IX)
Hexachloropropene (Appendix IX)
Isosafrole (Appendix IX)
Nitrobenzene-d5 (surrogate)

Internal Standard: Acenaphthene-d10

Target and Surrogates:

Hexachlorocyclopentadiene
2,4,6-Trichlorophenol
2,4,5-Trichlorophenol (not on PP list)
1-Chloronaphthalene (8270 C)
2-Chloronaphthalene
2-Nitroaniline (not on PP list)
Dimethyl phthalate
Acenaphthylene
3-Nitroaniline (not on PP list)
Acenaphthene
2,4-Dinitrophenol
4-Nitrophenol
Dibenzofuran (not on PP list)
2,4-Dinitrotoluene
2,6-Dinitrotoluene
Diethyl phthalate
4-Chlorophenylphenyl ether
Fluorene
4-Nitroaniline (not on PP list)
1-Naphthylamine (8270 C)
2-Naphthylamine (8270 C)
Pentachlorobenzene (8270 C)

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

TABLE 3 (cont.)

Analyte Quantitation and Internal Standards

1, 2, 4, 5-Tetrachlorobenzene (8270 C)
2, 3, 4, 6-Tetrachlorophenol (8270 C)
p-Phenylenediamene (Appendix IX)
Safrole (Appendix IX)
1,4-Naphthoquinone (Appendix IX)
Thionazine (Appendix IX)
5-Nitro-o-toluidine (Appendix IX)
1,2-Diphenylhydrazine (not on TCL list)
2-Fluorobiphenyl (surrogate)
2,4,6-Tribromophenol (surrogate)

Internal Standard: Phenanthrene-d10

Target and Surrogates:

4,6-Dinitro-2-methylphenol
N-Nitrosodiphenylamine
Diphenylamine (8270 C)
4-Bromophenylphenyl ether
Phenacetin (8270 C)
Hexachlorobenzene
4-Aminobiphenyl (8270 C)
Pentachlorophenol
Pentachloronitrobenzene (8270 C)
Pronamide (8270 C)
Phenanthrene
Anthracene
Di-n-butylphthalate
Carbazole (8270 B)
Fluoranthene
Sym-Trinitrobenzene (Appendix IX)
Diallate (Appendix IX)
4-Nitroquinoline-1-oxide (Appendix IX)
Methapyrilene (Appendix IX)
Isodrin (Appendix IX)

Internal Standard: Chrysene-d12

Target and Surrogates:

Benzidine (not on TCL list)
Pyrene
Butylbenzyl phthalate
3,3'-Dichlorobenzidine
p-Dimethylaminoazobenzene (8270 C)
Benzo (a) Anthracene
Bis (2-ethylhexyl) phthalate
Chrysene
3-Methylcholanthrene (8270 C)
Aramite (Appendix IX)
Chlorobenzilate (Appendix IX)
3,3'-Dimethylbenzidine (Appendix IX)
2-Acetylaminofluorene (Appendix IX)
Terphenyl-d14 (surrogate)

Internal Standard: Perylene-d12

Target and Surrogates:

Di-n-octyl phthalate
Benzo (b) fluoranthene
Benzo (k) fluoranthene
Benzo (a) pyrene
Indeno (1,2,3-cd) pyrene
Dibenz (a, h) anthracene
Dibenz (a, j) acridine (8270 C)
Benzo (ghi) perylene
7,12-Dimethylbenz (a) anthracene (8270 C)
Hexachlorophene (Appendix IX)

TABLE 4

PROCEDURE CONDENSATION

Clock

12 hours from injection of 50ng DFTPP.

Calibration Curve Criteria

RSD 20% for all compounds.
If not met:
Option 1) Linear least squares regression: $r \geq 0.995$
Option 2) Non-linear regression: coefficient of determination (COD) $r^2 \geq 0.99$ (6 points for second order)
Up to 10% of target analytes may be outside of the above criteria
Refer to section 7.5.2.1 for additional information.
Recommended minimum RF criteria for analytes listed in Table 6.

Continuing Calibration Check Criteria

All target analytes: $\leq$ 20%D
Recommended minimum RF criteria for analytes listed in Table 6.

Additional QC

LCS every extraction batch
MS/MSD every 20 samples

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

TABLE 5

CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

COMPOUND	PRIMARY ION	SECONDARY ION(S)
2-Picoline	93	66,92
Aniline	93	66,65
N-Nitrosodimethylamine	42	74,43
Phenol	94	65,66
Bis(2-Chloroethyl)ether	93	63,95
2-Chlorophenol	128	64,130
1,3-Dichlorobenzene	146	148,111
1,4-Dichlorobenzene	146	148,111
1,2-Dichlorobenzene	146	148,111
N-Nitrosomethylethylamine	88	42,43,56
Benzyl alcohol	108	77,79
2-Methylphenol	107	107,108,77,79,90
Bis(2-Chloroisopropyl)ether	45	77,121
4-Methylphenol	107	107,108,77,79,90
N-Nitroso-di-n-propylamine	70	42,101,130
Hexachloroethane	117	201,199
Nitrobenzene	77	123,65
Isophorone	82	95,138
2-Nitrophenol	139	65,109
2,4-Dimethylphenol	122	121,107
Benzoic acid	122	105,77
Bis(2-chloroethoxy)methane	93	95,123
2,4-Dichlorophenol	162	164,98
1,2,4-Trichlorobenzene	180	182,145
Naphthalene	128	129,127
4-Chloroaniline	127	129,65,92
Hexachlorobutadiene	225	223,227
4-Chloro-3-methylphenol	107	144,142
2-Methylnaphthalene	142	141
Hexachlorocyclopentadiene	237	235,272
2,4,6-Trichlorophenol	196	198,200
2,4,5-Trichlorophenol	196	198,97,132,99
2-Chloronaphthalene	162	164,127
2-Nitroaniline	65	92,138
Dimethyl phthalate	163	194,164
Acenaphthylene	152	151,153
3-Nitroaniline	138	108,92
Acenaphthene	153	152,154
2,4-Dinitrophenol	184	63,154
4-Nitrophenol	109	139,65
Dibenzofuran	168	139
2,4-Dinitrotoluene	165	63,89
2,6-Dinitrotoluene	165	89,63
Diethyl phthalate	149	177,150
4-Chlorophenylphenylether	204	206,141
Fluorene	166	165,167
4-Nitroaniline	138	92,108,65,80,39
4,6-Dinitro-2-methylphenol	198	105,51
N-Nitrosodiphenylamine	169	168,167
4-Bromophenylphenylether	248	250,141

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

TABLE 5 (cont.)

CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

COMPOUND	PRIMARY ION	SECONDARY ION(S)
Hexachlorobenzene	284	142,249
1,2-Diphenylhydrazine	184	77,92
Pentachlorophenol	266	264,268
Phenanthrene	178	179,176
Di-n-butyl phthalate	149	150,104
Carbazole	167	166,139
Fluoranthene	202	101,203
Benzidine	184	92,185
Pyrene	202	200,203
Butylbenzylphthalate	149	91,206
3,3-Dichlorobenzidine	252	254,126
Benzo(a)anthracene	228	229,226
Bis(2-ethylhexyl)phthalate	149	167,279
Chrysene	228	229,226
Di-n-octyl phthalate	149	167,43
Benzo(b)fluoranthene	252	253,125
Benzo(k)fluoranthene	252	253,125
Benzo(a)pyrene	252	253,125
Indeno(1,2,3-cd)pyrene	276	138,277
Dibenz(ah)anthracene	278	139,279
Benzo(ghi)perylene	276	138,277
N-Nitrosodiethylamine	102	42,57,44,56
N-Nitrosopyrrolidine	100	41,42,68,69
N-Nitrosomorpholine	56	116,86
Acetophenone	105	71,51,120
2,6-Dichlorophenol	162	63,98
α,α-Dimethylphenethylamine	58	91,65,134,42
N-Nitrosodi-n-butylamine	84	57,41,116,158
N-Nitrosopiperidine	114	42,55,56,41
O-toluidine	106	107,77,51,79
O,O,O-Triethylphosphorothioate	198	121,97,65
Hexachloropropene	213	211,215,117,106,141
Isosafrole	162	131,104,77,51
1-Chloronaphthalene	162	127,164
1-Naphthylamine	143	115,89,63
2-Naphthylamine	143	115,116
Pentachlorobenzene	250	252,108,248,215,254
1,2,4,5-Tetrachlorobenzene	216	214,179,108,143,218
2,3,4,6-Tetrachlorophenol	232	131,230,166,234,168
p-Phenylenediamene	108	80,53,54,52
Safrole	162	104,77,103,135
1,4-Naphthquinone	158	104,102,76,50,130
Thionazine	107	96,97,143,79,68
5-Nitro-o-toluidine	152	77,79,106,94
4-Aminobiphenyl	169	168,170,115
Diphenylamine	169	168,167
Pentachloronitrobenzene	237	142,214,249,295,265
Phenacetin	108	180,179,109,137,80
Pronamide	173	175,145,109,147
sym-Trinitrobenzene	75	213,120

TABLE 5 (cont.)

CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

COMPOUND	PRIMARY ION	SECONDARY ION(S)
Diallate	86	234,43,70
4-Nitroquinoline-1-oxide	174	101,128,75,116
Methapyrilene	97	50,191,71
Isodrin	193	66,195,263,265,147
p-Dimethylaminoazobenzene	225	120,77,105,148,42
7,12-Dimethylbenz(a)anthracene	256	241,239,120
3-Methylcholanthrene	268	252,253,126,134,113
Aramite	185	191,319,334,197,321
Chlorobenzilate	251	139,253,111,141
3,3'-Dimethylbenzidine	212	106,196,180
2-Acetylaminofluorene	181	180,223,152
Dibenz(a,j)acridine	279	280,277,250
Hexachlorophene	196	198,209,21,406,408
Phenol-d6 (surrogate)	99	42,71
2-Fluorophenol (surrogate)	112	64
2,4,6-Tribromophenol (surrogate)	330	332,141
Nitrobenzene-d5 (surrogate)	82	128,54
2-Fluorobiphenyl (surrogate)	172	171
Terphenyl-d14 (surrogate)	244	122,212
1,4-Dichlorobenzene-d4 (istd.)	152	115,150
Naphthalene-d8 (istd.)	136	68
Acenaphthene-d10 (istd.)	164	162,160
Phenanthrene-d10 (istd.)	188	94,80
Chrysene-d12 (istd.)	240	120,236
Perylene-d12 (istd.)	264	260,265

Primary ions must not be changed except in unusual instances where interference occurs with a co-eluting non-target analyte. In this case, a secondary ion may be used for quantitation with the following rules:

(1) The corresponding standard(s) (initial calibration curve and continuing calibration standard) must be re-quantitated with the secondary ion.

(2) Approval must be obtained from the Department Manager or the laboratory Operations Manager.

The quantitation ion must then be changed back to the one specified in Table 3 after quantitation of the samples(s).

Secondary ions are recommended only and may be changed depending upon instrument conditions (sensitivity, etc.). However, it is Katahdin policy that a minimum of 2 ions (primary and one secondary) be used for all GC/MS analyses.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

Table 6

RECOMMENDED MINIMUM RESPONSE FACTOR FOR INITIAL AND CONTINUING CALIBRATION

Semivolatile Compounds	Minimum Response Factor (RF)
Benzaldehyde	0.010
Phenol	0.800
Bis(2-chloroethyl)ether	0.700
2-Chlorophenol	0.800
2-Methylphenol	0.700
2,2'-Oxybis-(1-chloropropane)	0.010
Acetophenone	0.010
4-Methylphenol	0.600
N-Nitroso-di-n-propylamine	0.500
Hexachloroethane	0.300
Nitrobenzene	0.200
Isophorone	0.400
2-Nitrophenol	0.100
2,4-Dimethylphenol	0.200
Bis(2-chloroethoxy)methane	0.300
2,4-Dichlorophenol	0.200
Naphthalene	0.700
4-Chloroaniline	0.010
Hexachlorobutadiene	0.010
Caprolactam	0.010
4-Chloro-3-methylphenol	0.200
2-Methylnaphthalene	0.400
Hexachlorocyclopentadiene	0.050
2,4,6-Trichlorophenol	0.200
2,4,5-Trichlorophenol	0.200
1,1'-Biphenyl	0.010
2-Chloronaphthalene	0.800
2-Nitroaniline	0.010
Dimethyl phthalate	0.010
2,6-Dinitrotoluene	0.200
Acenaphthylene	0.900
3-Nitroaniline	0.010
Acenaphthene	0.900
2,4-Dinitrophenol	0.010
4-Nitrophenol	0.010
Dibenzofuran	0.800
2,4-Dinitrotoluene	0.200
Diethyl phthalate	0.010
1,2,4,5-Tetrachlorobenzene	0.010
4-Chlorophenyl-phenyl ether	0.400
Fluorene	0.900
4-Nitroaniline	0.010
4,6-Dinitro-2-methylphenol	0.010
4-Bromophenyl-phenyl ether	0.100
N-Nitrosodiphenylamine	0.010
Hexachlorobenzene	0.100

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

Table 6

RECOMMENDED MINIMUM RESPONSE FACTOR FOR INITIAL AND CONTINUING CALIBRATION (CONT.)

Semivolatile Compounds	Minimum Response Factor (RF)
Atrazine	0.010
Pentachlorophenol	0.050
Phenanthrene	0.700
Anthracene	0.700
Carbazole	0.010
Di-n-butyl phthalate	0.010
Fluoranthene	0.600
Pyrene	0.600
Butyl benzyl phthalate	0.010
3,3'-Dichlorobenzidine	0.010
Benzo(a)anthracene	0.800
Chrysene	0.700
Bis-(2-ethylhexyl)phthalate	0.010
Di-n-octyl phthalate	0.010
Benzo(b)fluoranthene	0.700
Benzo(k)fluoranthene	0.700
Benzo(a)pyrene	0.700
Indeno(1,2,3-cd)pyrene	0.500
Dibenz(a,h)anthracene	0.400
Benzo(g,h,i)perylene	0.500
2,3,4,6-Tetrachlorophenol	0.010

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

FIGURE 1

EXAMPLE OF RUNLOG LOGBOOK PAGE

KATAHDIN ANALYTICAL SERVICES
GC/MS SVOA INJ LOG INSTRUMENT: 5973-U

DATE OF DFTPP INJECTION: 010708

JOB	SAMPLE	DATAFILE	DF	ALS #	METHOD	UL INJ	CHEMIST	COMMENTS
	50 ng DFTPP	UD626	1	1	DFTPP3Q0	2.0	JW	OK
B	SSTD050UO107	UO563		2	US270C02	1.0		✓
	010	64		3				✓
	025	65		4				✓ Runs OK
	100	66		5				✓
	150	67		6				✓
	200	68		7				✓
A	SSTD 030 UO107	69(a)		8				✓ (a) = UTCLP02
	010	70(a)		9				✓ files
	025	71(a)		10				✓
	100	72(a)		11				✓
	150	73(a)		12				✓
	200	74(a)		13				✓
	8270 IND CHK	75(a)		14				

JW 010808

STANDARD	CODE
DFTPP	51176
CAL. STD.	51181 51182
IS MIX	STK 3505

REVIEWED AND APPROVED BY: _____
DATE: _____

QAMS364

0000015

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

FIGURE 2

EXAMPLE OF GC/MS STANDARDS RECEIPT LOGBOOK ENTRY

KATAHDIN ANALYTICAL SERVICES

STOCK STANDARDS RECEIVED

GCMS LABORATORY
REVIEWED BY/DATE:

AM P0946

AccuStandard® 125 Market St. · New Haven, CT 06513 · USA Tel. 203-786-5290 · www.accustandard.com
APP-9-176-D-20X
Pentachlorophenol
2.0 mg/mL in CH2Cl2
Lot: B3010100
Exp. Jan 10, 2013
1 mL
POISON
FOR LABORATORY USE ONLY
WARNING: This product contains a chemical(s) known to the State of California to cause cancer.
STORAGE Ambient
[redacted]

Recd
8/16/06
JLH

AMP0947

AccuStandard® 125 Market St. · New Haven, CT 06513 · USA Tel. 203-786-5290 · www.accustandard.com
APP-9-090-50X
4,6-Dinitro-o-cresol
5.0 mg/mL in MeOH
Lot: B1100296
Exp. Aug 16, 2012
1 mL
FLAMMABLE
FOR LABORATORY USE ONLY
STORAGE Ambient
[redacted]

AMP0948

AccuStandard® 125 Market St. · New Haven, CT 06513 · USA Tel. 203-786-5290 · www.accustandard.com
APP-9-145-50X
p-Nitrophenol
5.0 mg/mL in MeOH
Lot: B5050205
Exp. May 18, 2015
1 mL
POISON
FOR LABORATORY USE ONLY
STORAGE Ambient
[redacted]

QAMS294

0000045

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

FIGURE 3

EXAMPLE OF SVOA STANDARDS PREPARATION LOGBOOK ENTRY

GC/MS SVOA STANDARD PREP LOGBOOK

50863	8270 Stock (w/o MeOH)	3-15-06	7-7-06	JLK	AMP0844	8270 Megamix	300	2-22-07	4.2ml	150 ug/ml
					AMP0937	↓	300	3-15-07		
					AMP0911	APP IX #2	600	3-2-07		
					AMP0910	↓ 1	180	3-9-07		
					AMP0570	↓ 1	200	7-7-06		
					AMP0699	Organophosphat	300	8-14-06		
					AMP0934	Benzoic Acid	↓	3-9-07		
					AMP0907	Hexachlorophene		2-22-07		
					AMP0806	Benzidine		3-9-07		
					AMP0936	3,3'-Dichlorobenzidine	↓	3-14-07		
					AMP0932	8270 Surr	150	3-9-07		
					50861	DEA	300	3-13-07		
					BY3E90	MeCl2	580	~		
50864	8270 level 1	3-15-06	7-7-06	JLK	50863	8270 Stock	70	7-7-06	1.05ml	10 ug/ml
					BY3E90	MeCl2	980			
50865	8270 level 2	3-15-06	7-7-06	JLK	50863	8270 Stock	150	7-7-06	0.90ml	25 ug/ml
					BY3E90	MeCl2	750			
50866	8270 level 3	3-15-06	7-7-06	JLK	50863	8270 Stock	600	7-7-06	1.8ml	50 ug/ml
					BY3E90	MeCl2	1200			
50867	8270 level 4	3-15-06	7-7-06	JLK	50864	8270 Stock	700	7-7-06	1.05ml	100 ug/ml
					BY3E90	MeCl2	350			

Reviewed by/Date:

CAMS284

0000057

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8081

Prepared By: Peter Lemay Date: 7/96

Approved By:

Group Supervisor: Peter Lemay Date: 1/15/01

Operations Manager: John C. Burton Date: 1/15/01

QA Officer: Deborah J. Nadeau Date: 1.22.01

General Manager: Dennis C. Kuhnke Date: 1/16/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
CR 8081A	Format changes, added pollution prevention, minor changes to sections 7, 8 and Table 1.	DN	1.22.01	1/22/01
03 8081A	Changes to comply with South Carolina requirements - added linear calibration option, retention time window criteria & other minor changes to surrogate criteria.	DN	5.21.01	5.21.01
04 8081A	Changed to practice of reporting higher value. Other minor changes to Table 1 & 2, section 7.5.3 and section 7.4.3.	DN	5.21.02	5.21.02
05 8081A	Added definitions and information for the new data processing system. Replaced several figures with updated ones.	MRC	05.04.04	05.04.04
06 8081A	added alternative CV Conc. changed data checklist minor changes throughout added wording to section 8	LAD	3 05/05 LAD	3 05/05 6-13-06

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8081

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
07	Added retention time window criteria. Sect. 7.4.2 - added to Shake samples before vialing.	LAD	03/06	03/06
08	Sect. 1.4 - Updated to include waste streams. Sect. 4.5 - removed balance Changed makeup gas from N_2 to Ar Me. Changed T.C. from 5 to 2 peaks. Updated column confirmation. Changed corr. coef. to coeff. of determination	LAD	06/07	06/07
09	Added extraction method 3535 for AQ Samples Updated method references. Added Katahdin Analytical Environmental Health and Safety Manual. Changed number of peaks quantitated for Toxaphene to 3 or 5. Added S.C. clarification on marginal exceedences.	LAD	02/09	02/09
10	CHANGES made to Sections 4.1, 7.4, 7.5, 8.0 and 10.0 for COMPLIANCE with DoD QSM version 4.1	LAD	08/09	08/09
11	Added Table 2 with DoD QSM version 4.1 QC Requirements. Updated Figure 1 and 2.	LAD	04/10	04/10

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8081

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-302-11**, titled **ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8081**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-302-11**, titled **ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8081**.

Recipient: _____ Date: _____

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8081

1.0 SCOPE AND APPLICATION

This SOP describes all aspects of the analysis of extracts of solid and aqueous samples for Pesticides by EPA Method 8081B, as performed by Katahdin Analytical Services, Inc. including sample analysis, data review, standard preparation and instrument calibration.

It is applicable to the following compounds: aldrin, alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, chlordane, 4,4'-DDT, 4,4-DDE, 4,4'-DDD, dieldrin, endosulfan I, endosulfan II, endosulfan sulfate, endrin, endrin aldehyde, heptachlor, heptachlor epoxide, toxaphene, endrin ketone, and methoxychlor. Extracts are analyzed by Gas Chromatography-Electron Capture Detector (GC-ECD).

1.1 Definitions

ANALYTICAL BATCH: 20 or fewer samples which are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods.

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, laboratory reagent grade water is used as a blank matrix; however a universal blank matrix does not exist for solid samples, and therefore, no matrix is used. The blank is taken through the appropriate steps of the process.

INDEPENDENT CALIBRATION VERIFICATION (ICV): A verification of the ratio of instrument response to analyte amount. ICV solutions are prepared from stock solutions which are independent from the stock solutions used to prepare the calibration standards.

CALIBRATION STANDARD (WORKING STANDARD): A solution prepared from the stock standard solution, which is used to calibrate the instrument response with respect to analyte concentration.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

STANDARD CURVE (CALIBRATION CURVE): A curve that plots concentration of known analyte standard versus the instrument response to the analyte.

STOCK STANDARD SOLUTION: A concentrated solution containing a single certified standard that is a method analyte, or a concentrated solution of a single analyte prepared in the laboratory with an assay reference compound. Stock standard solutions are used to prepare calibration standards.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

KATAHDIN INFORMATION MANAGEMENT SYSTEM (KIMS) : A complete multi-user system with the capabilities of integrating laboratory instrumentation, generating laboratory worksheets, providing complete Lab Order status and generating reports. KIMS utilizes these features through a database.

PE NELSON TURBOCHROM: A data acquisition system that is used to collect chromatographic data. The system can also be used to archive raw data files.

HP ENVIROQUANT: A data acquisition system that is used to collect chromatographic data.

TARGET: A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

TARGET DB: An oracle database used to store and organize all Target data files.

QUICKFORMS: A laboratory reporting software for Target and Target DB. The QuickForms report module for Target is preconfigured with generalized forms and US EPA CLP report forms and disk deliverables, which can be customized.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of pesticides by method 8081, current revision. Each analyst must demonstrate the ability to generate acceptable results with this method.

It is the responsibility of all Katahdin technical personnel involved in analysis by method 8081, current revision, to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be

recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Health and Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Analytical Environmental Health and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Wastes generated during standards preparation are disposed of in the Mixed Flammable Waste (O). After the extracts have been analyzed, the autosampler vials and any expired standard vials or ampules are disposed of in the Organic Vial Waste (P).

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8081

2.0 SUMMARY OF METHOD

2.1 Method 8081 provides gas chromatographic conditions for the detection of ppb concentrations of certain organochlorine pesticides. Prior to the use of this method, appropriate sample extraction techniques must be used. Both neat and diluted organic liquids (Method 3580, waste dilution) may be analyzed by direct injection. A 2-5 ul aliquot of sample is injected into a gas chromatograph (GC) using the direct injection technique, and compounds in the GC effluent are detected by an electron capture detector (ECD).

2.2 The sensitivity of Method 8081 usually depends on the concentration of interferences rather than on instrumental limitations. If interferences prevent detection of the analytes, Method 8081 may also be performed on samples that have undergone cleanup. Method 3660, Sulfur Cleanup, by itself or in conjunction with Method 3620, Florisil Column Cleanup, may also be used to eliminate interferences in the analysis.

3.0 INTERFERENCES

3.1 Interferences by phthalate esters can pose a problem in pesticide determinations when using the electron capture detector. Common flexible plastics contain various amounts of phthalates. Care has to be taken to avoid using any plastic materials during the extraction process. Exhaustive cleanup of reagents and glassware may be required to eliminate background phthalate contamination.

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph

4.1.1 GC Hewlett Packard 6890 or 5890 series I or II or 6890 connected to the Turbochrom or Enviroquant data system,or equivalent.

4.1.2 Columns: Instruments are configured with a pre-column originating from the injection port which is connected to deactivated glass Y splitter that connects two different columns to two detectors. The most commonly used columns are: RTX-35 30M x 0.53 mm ID, RTX-5 30M x 0.53 MM ID, or RTX-1701 30M x 0.53 mm ID. Equivalent columns can be used.

4.1.3 Detectors: Electron capture detectors (ECD).

4.2 Volumetric flasks, class A: sizes as appropriate with the ground-glass stoppers.

4.3 Syringes: various sizes for preparing standards and injecting samples on the instrument.

4.4 Vials: various sizes and types including crimp tops.

4.6 Refrigerator for storage of extracts and standards.

5.0 REAGENTS

5.1 Solvents

5.1.1 Hexane: pesticide quality or equivalent for diluting samples and standards.

5.2 Standards

5.2.1 Stock standard solutions: Solutions purchased from suppliers like Restek or other acceptable retailers. Expiration dates are one year from date of opening vial or sooner if manufacturers date is less. Upon receipt, all standards are logged into the appropriate logbook with the date of receipt, expiration date, source, lot number, solvent and concentration of compounds.

5.2.2 Calibration standards: Prepared through the dilution of the stock standards with hexane. Expiration date is 6 months or sooner. Information is documented in a separate logbook.

5.2.3 Pesticide Working standards: Prepared by diluting the stock mix of 2000 ug/ml that contains all single component pesticides into hexane to give final concentrations of: 0.005, 0.01, 0.025, 0.05, 0.10, and 0.25 ug/ml. The mix, referred to as INDAB, also contains two surrogates: Tetrachloro-m-xylene and Decachlorobiphenyl, which are at the same concentrations as the pesticides.

5.2.4 Independent Calibration Verification Standard: Prepared as above using a standard independent of the calibration standards.

5.2.5 Multicomponent Pesticide Working standards: Toxaphene is prepared by diluting the Toxaphene stock solution to a concentration of 1.0 ug/ml. Technical chlordane is prepared similarly except to a concentration of 0.50 ug/ml.

5.2.6 Evaluation Mix: Prepared by diluting the stock solution to a concentration of 0.20 ug/mL.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Extracts must be stored under refrigeration and analyzed within 40 days of extraction.

7.0 PROCEDURES

EXTRACTION - Refer to the appropriate SOP for the correct extraction procedure. In general, water samples are extracted using methods 3510, 3520 or 3535 while solid samples use methods 3540, 3545 or 3550.

7.1 INSTRUMENT CONDITIONS

Refer to the instrument logbook for the current column and conditions.

Typical conditions are:	Makeup flow:	60 ml/min Nitrogen or Ar/Methane
	Column flow:	3.75 ml/min
	Injector Temp:	200
	Detector Temp:	300
	Oven Ramp:	160(0) - 5/min - 260(10)
	Run time:	24 min
	Injection size:	2 uL

7.2 CALIBRATION

7.2.1 The GC system is calibrated using the external standard calibration procedure. A six-point calibration standard mix of the INDAB mix listed in Reagents Section 5.2.2 is prepared along with a single point standard of Toxaphene and Technical Chlordane.

If the sample contains Toxaphene, a six-point calibration curve is analyzed. If the sample contains Chlordane and the analysis request is for Technical Chlordane, a six-point calibration curve is analyzed. If the analytical request is for the two components alpha-Chlordane and gamma-Chlordane, these two compounds are quantitated from the INDAB mix.

Toxaphene is calibrated using the 5 to 10 major peaks of the standard. The Target system will calculate a peak height for all 5 to 10 peaks. A calibration curve is prepared in Target using the peak heights of the 5 to 10 peaks against the concentration of the standard.
Technical Chlordane is calibrated using 3 to 5 major peaks of the standard. The Target system will calculate a peak height for all three to five peaks. A separate calibration curve for each of the 3 to 5 peaks is prepared in Target using the peak height against the concentration of the standard.

Each calibration standard is injected using the technique that is used to introduce the actual samples into the GC. The Target system will calculate a peak height for each compound. A calibration curve can be prepared in Target using the peak height against the concentration of the standard. A non-linear calibration applying a second order polynomial (quadratic fit) equation is used

to prepare the curve. In order to be used for quantitative purposes, the Coefficient of Determination must be greater than or equal to 0.990. The quadratic equation is:

$$y = ax^2 + bx + c$$

where: y = Instrument response
b = Slope of the line
x = Concentration of the calibration standard
c = The intercept

Please note that a non-linear calibration model may not be allowable for certain states, federal programs, or clients. South Carolina does not allow non-linear calibration for compliance work originating in their state. In these cases, a linear calibration model must be used. The linear equation is

$$y = bx + c$$

where: y = Instrument response
b = Slope of the line
x = Concentration of the calibration standard
c = The intercept

The calibration curve is calibrated the same way as the second order polynomial equation except that a five-point calibration standard mix is used.

7.2.2 The INDAB mix calibration curve must be checked initially by analyzing a standard containing the same analytes as the curve but prepared from another source. If the response of the analytes from the independent source varies by more than $\pm$ 20%, a new independent source standard must be analyzed or a new calibration curve must be prepared and/or analyzed.

7.2.3 The working calibration curve must be verified on each 12-hour shift that samples are to be analyzed by injecting the mid-point calibration standard.

7.3 RETENTION TIME WINDOWS

7.3.1 Three injections of all single component standard mixtures and multiresponsive products throughout the course of a 72-hour period.

7.3.2 The standard deviation of the three retention times is calculated for each single component standard. For multiresponsive products, a major peak from the envelope is chosen and a standard deviation is calculated using the three retention times for that peak.

7.3.3 Plus or minus three times the standard deviation of the retention times for each standard is used to define the retention time window; however, the experience of the analyst should weight heavily in the interpretation of chromatograms. For multiresponsive analytes, the analyst should use the retention time window, but should primarily rely on pattern recognition.

7.3.4 Retention time windows are calculated for each standard on each GC column and whenever a new GC column is installed. The data is kept on file in the laboratory.

7.3.5 If the calculated retention time window results in a value of 0.03 minutes or less, the laboratory will apply nominal windows. This is done in order to avoid any false negative hits because of the window being too narrow. The windows are: ± 0.05 for Heptachlor, Aldrin and all BHC compounds, ± 0.07 for all other target analytes. By utilizing these windows, a false positive hit may be initially indicated, but an experienced analyst could determine a false positive by carefully evaluating the chromatograms. Please note that the use of nominal retention time windows may not be allowable for certain states, federal programs, or clients. South Carolina does not allow the use of nominal limits for compliance work originating in their state. In these cases, a window of ± 0.03 minutes must be used if the established retention time window is less than 0.03 minutes.

7.4 GAS CHROMATOGRAPHIC ANALYSIS

7.4.1 Before calibration is performed, and at the beginning of each 12 hour shift, the system is evaluated for analyte degradation by the analysis of a standard mix containing only endrin and 4,4'-DDT, often called an evaluation mix (EVAL):

COMPOUND	CONCENTRATION
Endrin	0.20 ng/uL
DDT	0.20 ng/uL

The % breakdown of DDT and the % breakdown of Endrin is calculated using the following formulas (PH = Peak Height):

$$\%\ \text{Breakdown DDT} = \frac{(\text{PH [DDD]} + \text{PH [DDE]})}{(\text{PH [DDD]} + \text{PH [DDE]} + \text{PH [DDT]})} * 100$$

$$\%\ \text{Breakdown Endrin} = \frac{(\text{PH [Endrin Aldehyde]} + \text{PH [Endrin Ketone]})}{(\text{PH of [Endrin Aldehyde]} + \text{PH of [Endrin Ketone]} + \text{PH of [Endrin]})} * 100$$

The breakdown of either DDT or Endrin in the evaluation mix cannot exceed 15%. If there is breakdown of either compound exceeding 15% before starting a calibration, instrument maintenance must be performed. A calibration can not be run until the

evaluation mix meets the acceptance criteria. If the exceeding breakdown occurs during the analysis sequence, then any samples analyzed after a failing evaluation mix must be reanalyzed. Reanalysis can not resume until after an acceptable evaluation mix.

7.4.2 Gently shake sample extracts before vialing for analysis.

7.4.3 All instrument injections are performed using the direct injection technique with an autosampler set for 2-5 uL injection volumes.

7.4.4 Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration as listed in section 7.2 followed by sample extracts interspersed with mid-concentration calibration standards. Before any samples are analyzed the instrument must be calibrated by analyzing a six-point calibration or a 0.05ppm concentration standard (calibration verification standard). If a CV is run, the calculated concentration must not exceed a difference of ± 15%. DoD allows a difference of ± 20%. Each sample analysis must be bracketed with an acceptable initial calibration and closing CV or an opening CV and a closing CV for each 12-hour shift. The closing CV standard is at 0.25ppm. The calibration standard must also be injected at intervals of not less than once every ten samples and at the end of the analysis sequence. If the CV fails, the instrument is checked for any obvious problems and maintenance is performed if deemed necessary. All samples that were injected after the last standard that last met the QC criteria must be evaluated to prevent mis-quantitations and possible false negative results, and re-injection of the sample extracts may be required. However, if the standard analyzed after a group of samples exhibits a response for an analyte that is above the acceptance limit, i.e. >15%, and the analyte was not detected in the specific samples analyzed during the analytical shift, then the extracts for those samples do not need to be reanalyzed, as the CV standard has demonstrated that the analyte would have been detected were it present. In contrast, if an analyte above the QC limits was detected in a sample extract, then re-injection is necessary to ensure accurate quantitation. If an analyte was not detected in the sample and the standard response is more than 15% below the initial calibration response, then re-injection is necessary to ensure that the detector response has not deteriorated to the point that the analyte would not have been detected even though it was present.

7.4.5 The center of the retention time window for each analyte and surrogate is established by using the absolute retention time for each analyte and surrogate from the daily opening calibration verification or initial calibration.

7.4.6 The identification of Pesticides is based on agreement between the retention times of peaks in the sample chromatogram with the retention time windows established through the analysis of standards of the target analytes. An analyte

is tentatively identified when a peak from a sample falls within the absolute retention time window. Each tentative identification must be confirmed using a second GC column of dissimilar stationary phase or using another technique such as GC/MS. If the retention times of the peaks on both columns fall within the retention time windows on the respective columns, then the target analyte identification has been confirmed.

7.4.7 If the response for an analyte exceeds the calibration range of the system, the sample must be diluted and reanalyzed.

7.4.8 If peak detection and identification are prevented due to interferences, the hexane extract may need to undergo a cleanup. The extract may be subjected to a florisil cleanup (method 3620) and/or a sulfur cleanup (method 3660). Whenever a sample receives a cleanup, the associated QC must also be subjected to the same cleanup(s) and reanalyzed.

7.4.9 When a GC system is determined to be out of control because either a CV can not pass or a six point calibration does not meet the coefficient of determination criteria, instrument maintenance is likely necessary. Routine instrument maintenance may involve changing the septum, replacing the liner, clipping the pre-column, replacing the Y connector, or replacing the column. This information is recorded in the instrument run log (Figure 1). When an instrument requires more severe maintenance like replacing the ECD or an electronic board, this information is written in the instrument maintenance logbook. Refer to Katahdin SOP CA-101, Equipment Maintenance.

7.4.10 The concentration of an analyte is calculated by using the calibrated curve that is prepared in Target. When an analyte is identified, Target displays a concentration after the file is processed through the appropriate calibrated method.

7.4.11 The concentrations from the reports are then incorporated with the extraction data to arrive at a final concentration.

7.4.11.1 Water: Concentration (ug/L) = (C) (Vt)/(Vs)

7.4.11.2 Soil / Sediment:Concentration (mg/kg) = (C) (Vt)/(Ws) (D)

where, C = concentration calculated by Target in ug/ml
Vt = Volume of total extract including any instrument dilutions
Vs = Volume of sample extracted
Ws = Weight of sample extracted
D = Decimal total solids

7.5 Data Review

7.5.1 Initial Data Review

The initial data review is accomplished by the analyst who ran the samples. This review is of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed. The initial data review is performed on the detailed quantitation reports of the analyzed samples. This data review examines criteria that directly impact whether or not the sample needs to be reanalyzed and/or extracted. These criteria include:

- QC criteria for method blank, LCS, MS/MSD, and calibration – refer to section 8.0.
- Surrogate recovery
- Chromatography: cleanups, manual integration.
- Target compound detection: quantitation, confirmation, false positives.

The requirement of the GC laboratory is that this initial data review be completed no later than the end of the next workday. After the analyst has completed his or her initial data review, the information is then ready to be processed for reporting. Refer to section 7.7.

7.5.2 Surrogate recovery

All recoveries must meet the most recently laboratory established acceptance limits, which are listed on the Laboratory Surrogate Acceptance Limit sheet. For DoD work, the surrogates must meet the acceptance limits in the DoD QSM.

The sample is evaluated for recoveries of the two surrogates. The recoveries of both surrogates are evaluated on both the primary and secondary column. The higher recovery from both columns is reported on the analytical report for both surrogates. The sample chromatogram is reviewed for any interferences before determining whether to accept a sample based on the surrogate recoveries. If the surrogate recovery is affected by matrix interference, the sample result may be accepted with narration. If the recovery of one surrogate is outside of the laboratory established acceptance limit on one or both columns, and the second is acceptable, the data is narrated. If the recoveries for both surrogates are not acceptable because the recoveries are high and the sample does not contain any analytes above the PQL, the data is narrated. If the recoveries for both surrogates are low and there is no apparent matrix effect, the sample is reextracted.

For method blanks, if the recoveries of both surrogates are low or high, and the blank does not contain any target analytes above the PQL, and the

recoveries of both surrogates in the sample(s) are acceptable, the data is narrated. If the recoveries in the blank are low and it does not contain any target analytes above the PQL, and the recoveries in the samples are acceptable but the sample contains one or more target analytes above the PQL, the sample may be reextracted.

For laboratory control samples (LCS), if the only discrepancy in the extraction batch is with the LCS, and the analyte spike recoveries are acceptable, the data is narrated. If the recoveries of both the surrogates and the analyte spikes are low, the samples may need to be reextracted.

For DoD work, Q-flag all detected analytes in the sample if the surrogates fail the acceptance criteria.

7.5.3 Chromatography

The chromatography should be examined for the presence of any non-target peaks, which can be used as an indication of whether or not matrix interference might be influencing surrogate recoveries. If the chromatogram indicates interferences, then a cleanup may be needed. See section 7.4.7.

Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating the peak of concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.

Each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. Should a manual integration be necessary (for instance, due to a split peak, peak tailing, or incomplete resolution of isomeric pairs), an “m” qualifier will automatically be printed on the quantitation report summary. The analyst will date and initial the “m” on the quanitation report summary and assign a code that indicates the reason for the manual integration. Refer to Katahdin SOP QA-812 “Manual Integration on GC/MS, GC, HPLC and IC Datasystems” for more information.

7.5.4 Target Compound Detection

GC analysis relies heavily on the experience of the analyst. Sample chromatograms must be evaluated focusing on scientific judgment, knowledge of the column behavior and matrix effects. The chromatogram from channel A is evaluated with that from channel B. If a target analyte is present on both channels and the concentration is within the calibration range, and the quantitation from both chromatograms agrees within $\pm$40%, the analyte is considered to be present in the sample. In cases where the

RPD is greater than 40% and the analyte is reported, the analyte must be J-flagged indicating that the result is an estimated value. The higher of the two concentrations is reported unless matrix interference is causing erroneously high results. In this case report the lower result and narrate. Sometimes interference on one column (i.e. sulfur) will prevent a target analyte from detection and it is present on the conformational column. In this scenario, the result would be reported from one column and need to be "Q" flagged to indicate that it was not confirmed on a second column.

All flagged data must be discussed in the narrative

In order to avoid reporting false positives, identified peaks on a chromatogram may need to be undetected electronically in Target. The possible scenarios are: If an analyte is present on one column but its concentration is below the PQL, if an analyte is present on one column but does not confirm on the other channel, or if an analyte is present but its retention time is ±0.04 minutes or more than the retention time of the analyte in the preceding CV.

The GC Analyst must rely on technical experience in reviewing chromatograms in determining if a hit is an actual analyte or a false positive.

7.6 Reporting

After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into QuickForms. Depending on the QC level requested by the client, a Report of Analysis (ROA) and additional reports, such as LCS forms and chronology forms, are generated. The package is assembled to include the necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded to the secondary reviewer. The secondary reviewer validates the data and checks the package for any errors. When completed, the package is sent to the department manager for final review. A completed review checklist (Figure 2) is provided with each package. The final data package from the Organics department is then processed by the Data Management department.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 and to details in this section for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The criteria does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in this section or in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is

suspect. The corrective actions listed in this section and in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The Department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 For each analytical batch (up to 20 samples), a method blank, laboratory control sample (LCS), matrix spike and matrix spike duplicate are analyzed. They are carried through all stages of the sample preparation and analysis steps.

8.2 Spike concentrations: The LCS and the MS/MSD are spiked with the twenty single component pesticides at the same concentration. The spike concentrations are:

	WATER ug/L	SOILS ug/Kg
Pesticides	0.50	16.7

The surrogate spike concentrations in the final extract are:

	WATER ug/L	SOILS ug/Kg
Tetrachloro-m-xylene(TCX)	1.0	33.3
DCB	1.0	33.3

8.3 LCS and MS/MSD acceptance criteria and Corrective Action: All QC samples are calculated for percent recovery of the spiked analyte(s). The recoveries are compared to laboratory established acceptance limits. Refer to Katahdin SOP QA-808, "Generation and Implementation of Statistical QC Limits and/or Control Charts," current revision. For DoD work, the recoveries are compared to DoD QSM acceptance limits.

The LCS recoveries for all analytes are evaluated. All of the compounds of interest must fall within the established statistical limits with the following sporadic exceedance allowances. **South Carolina does not allow for marginal exceedances for compliance work originating in their state.**

Number of Analytes	Number of Allowable Exceedances
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
<11	0

If less than the number of allowable exceedances fail the statistical limits, no corrective action is needed. If greater than the number of allowable exceedances fail the statistical limits, corrective action may be taken. Corrective actions may vary with each situation. However, in the case where the failures are high and the samples are non-detect for those compounds, then no corrective action is required. Otherwise, corrective action may involve reanalysis or recalibration. The specific corrective actions taken will rely on analyst experience to make sound scientific judgments while considering client objectives, other quality control indicators and/or the ability to reanalyze a sample within holding time.

If a spike compound is outside of the acceptance limits in the matrix spike sample but is acceptable in the LCS, the data is considered acceptable. The cause of the failure is possibly attributable to matrix interference. However, if the compound fails in both the LCS and the MS/MSD, the result for that analyte is suspect and may not be reported for regulatory compliance purposes.

Please note that established acceptance limits that are wider than 70-130% may not be allowable for certain states, federal programs, or clients. For South Carolina, the acceptance limits for the spiked analytes will be 70-130% or narrower.

DoD work requires Q-flagging the specific LCS analytes that fail and are detected in the associated samples. MS/MSD failures require a J-flag in the parent sample for the analytes that fail the acceptance criteria.

8.4 Surrogate acceptance criteria and Corrective Action: Surrogate recoveries are calculated on all samples, blanks and spikes. The recoveries are compared to laboratory established acceptance limits.

When a sample has a surrogate that falls outside of the laboratory established acceptance limit window, the problem should be investigated. If the recovery looks like it is affected by the sample matrix, the sample may be reinjected to confirm matrix interference. When a sample has no detectable surrogate recovery, the sample should be reextracted.

For DoD work, Q-flag all detected analytes in the sample if the surrogates fail the acceptance criteria.

8.5 CAR: Whenever data is not acceptable because of a failing LCS or surrogate recovery, a corrective action report (CAR) must be initiated as soon as possible.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined prior to sample analysis per type of instrument and filed with the Organic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revision of Method 8081 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition. Final Update IV, dated February, 2007, Method 8081B.

Department of Defense Quality Systems Manual for Environmental Laboratories (DoD QSM), Version 4.1, 04/22/09.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

Katahdin Analytical Services, Inc., SOP CA-106, Standard Preparation, Documentation and Traceability.

Katahdin Analytical Services, Inc., SOP CA-515, Preparation of Aqueous Samples for Pesticides/PCBs Analysis-Methods 3510 and 3520.

Katahdin Analytical Services, Inc., SOP CA-500, Preparation of Soil/Sediment Samples by Sonication Using Method 3550 for Subsequent Pesticides/PCBs Analysis.

Katahdin Analytical Services, Inc., SOP CA-524, Preparation of Soil/Sediment Samples by Soxhlet Extraction Using Method 3540 for Subsequent Pesticides/PCBs Analysis.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8081

LIST OF FIGURES

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8081

TABLE 1

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Method blank	One per prep batch of twenty or fewer samples	No analyte detected >PQL DoD: no analyte detected >1/2 PQL and >1/10 the amount measured in sample	(1) Investigate source of contamination (2) Evaluate the samples and associated QC:i.e. If the blank results are above the PQL, report sample results which are < PQL or > 10X the blank concentration. Otherwise, reprep a blank and the remaining samples.
LCS	One per prep batch of twenty or fewer samples	Statistically derived limits. Note that limits wider than 70-130% are not allowable for some states, programs or clients, i.e. South Carolina. See also section 8.4 of this SOP for more information on allowable exceedances	(1) Evaluate the samples and associated QC: i.e. If an MS/MSD was performed and acceptable, narrate. If an LCS/LCSD was performed and only one of the set was unacceptable, narrate. If the surrogate recoveries in the LCS are low but are acceptable in the blank and samples, narrate. If the LCS recovery is high but the sample results are < PQL, narrate. Otherwise, reprep a blank and the remaining samples.
CCV	If calibration curve previously analyzed, analyze daily before samples and after every 10 samples.	$\pm$ 15% D	(1) Evaluate the samples: If the %D>+15% and sample results are <PQL, narrate. If %D>$\pm$15% only on one channel, narrate. If %D>$\pm$15% for the closing CV, and is likely a result of matrix interference, narrate. Otherwise, reanalyze all samples back to last acceptable CV.
Matrix Spike\ Matrix Spike Duplicate	One for every set of 20 samples	Same as for LCS	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS/MSD are unacceptable, reprep the samples and QC.
6 pt of INDAB mix with mid-pt cal of Toxaphene and Chlordane	Initial cal prior to sample analysis	6pt calibration coefficient of determination $\geq$ 0.990	(1) Repeat Initial calibration (2) If single pt cal Toxaphene, or Chlordane is identified in analysis of sample, 6 pt calibration run of identified compound with reanalysis of sample.
Independent calibration verification	Once after Initial calibration	± 20 % D	Reanalyze standard Reprep standard Reprep standard from fresh stock.
Demonstrate ability to generate acceptable P & A using 4 replicate analyses of a QC check standard	One time per analyst initially and annually thereafter.	All recoveries within method QC acceptance limits	Recalculate results; locate and fix problem; rerun P & A study for those analytes that did not meet criteria prior to sample analysis
MDL study	Refer to KAS SOP QA-806, “Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications”, current revision.		

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8081

TABLE 2

DoD QSM VERSION 4.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria.	Not Applicable (NA).	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification	Refer to current revision of SOP QA-806				
LOQ establishment and verification	Refer to current revision of SOP QA-806				
Retention time (RT) window width calculated for each analyte and surrogate	At method set-up and after major maintenance (e.g., column change).	RT width is ± 3 times standard deviation for each analyte RT from a 72-hour study.	NA.	NA.	
Breakdown check (Endrin / DDT Method 8081 only)	At the beginning of each 12-hour period, prior to analysis of samples.	Degradation ≤ 15% for both DDT and Endrin.	Correct problem then repeat breakdown check.	Flagging criteria are not appropriate.	No samples shall be run until degradation ≤ 15% for both DDT and Endrin.
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	One of the options below: Option 1: RSD for each analyte ≤ 20%; Option 2: linear least squares regression: r ≥ 0.995; Option 3: non-linear regression: coefficient of determination (COD) r2 ≥ 0.99 (6 points shall be used for second order). Mid point calibration of toxaphene and chlordane; if detected in sample, 6-point calibration is performed.	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. Calibration may not be forced through the origin. Quantitation for multicomponent analytes such as chlordane, or toxaphene must be performed using a 5-point calibration. Results may not be quantitated using a single point.
Retention time window position establishment for each analyte and surrogate	Once per ICAL and at the beginning of the analytical shift.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8081

TABLE 2

DoD QSM VERSION 4.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Second source calibration verification (ICV)	Immediately following ICAL.	All project analytes within established retention time windows. GC methods: All project analytes within ± 20% of expected value from the ICAL	Correct problem, rerun ICV. If that fails, repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
calibration verification (CCV)	Prior to sample analysis, after every 10 field samples, and at the end of the analysis sequence.	All project analytes within established retention time windows. GC methods: All project analytes within ± 20% of expected value from the ICAL	Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed. Retention time windows are updated per the method.
Method blank	One per preparatory batch.	No analytes detected > ½ RL (> RL for common lab contaminants) and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Reprepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result. Contact Client if samples cannot be reprepped within hold time.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory control sample (LCS) containing all analytes to be reported, including surrogates	One per preparatory batch.	The laboratory shall use laboratory control limits (CLs) or use DoD-generated LCS-CLs, if available depending on project requirements. In-house CLs may not be greater than ± 3 times the standard deviation of the mean LCS recovery. A number of analytes may fall outside the CL but within marginal exceedance limit depending on the total number of analytes in the LCS.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. Refer to Table G-1 for number of marginal exceedences allowed. Contact Client if samples cannot be reprepped within hold time.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8081

TABLE 2

DoD QSM VERSION 4.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix spike (MS)	One per preparatory batch per matrix if sufficient sample is available.	For matrix evaluation, use laboratory LCS CLs or use DoD-generated LCS-CLs, if available depending on project requirements.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix Spike duplicate (MSD)	One per preparatory batch per matrix if sufficient sample is available.	MSD: For matrix evaluation, use laboratory LCS CLs or use DoD-generated LCS-CLs, if available depending on project requirements. MS/MSD: RPD ≤ 30%.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Surrogate spike	All field and QC samples.	The laboratory shall use laboratory surrogate CLs or use DoD-generated surrogate CLs, if available depending on project requirements.	For QC and field samples, correct problem then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary. Contact Client if samples cannot be reprepped within hold time.	Apply Q-flag to all associated analytes if acceptance criteria are not met.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Confirmation of positive results (second column or second detector)	All positive results must be confirmed	Calibration and QC criteria same as for initial or primary column analysis. Results between primary and second column RPD ≤ 40%.	NA.	Apply J-flag if RPD > 40%. Discuss in the case narrative.	Use project-specific reporting requirements if available; otherwise, use method reporting requirements; otherwise, report the result from the primary column (see Box D-16).
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8081

TABLE 3

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-302-11	METHOD 8081, current revision
Apparatus/ Materials	None	
Reagents	None	
Sample preservation/ handling	None	
Procedures	7.3.5 If the calculated retention time window results in a value of 0.03 minutes or less, the laboratory will apply nominal windows. This is done in order to avoid any false negative hits because of the window being to narrow. The windows are: ± 0.05 for Heptachlor, Aldrin and all BHC compounds, ± 0.07 for all other target analytes. By utilizing these windows, a false positive hit may be initially indicated, but an experienced analyst could determine a false positive from scrutinizing the chromatograms. Please note that the use of nominal retention time windows may not be allowable for certain states, federal programs, or clients. South Carolina does not allow the use of nominal limits for compliance work originating in their state. In these cases, a window of ± 0.03 minutes must be used if the established retention time window is less than 0.03 minutes.	7.6.3 If the standard deviation of the retention times for a target compound is 0.000 (i.e., no difference between the absolute retention times), then the laboratory may either collect data from additional injections of standards or use a default standard deviation of 0.01 minutes. (Recording retention times to three decimal places rather than only two should minimize the instances in which the standard deviation is calculated as 0.000).
QC - Continuing Calibration	None	
QC - LCS	None	
QC - Accuracy/Precision	None	
QC - MDL	PQL – Practical Quantitation Level – three to ten times the MDL.	EQL – Estimated Quantitation Level – five to ten times the MDL

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8081

FIGURE 1

EXAMPLE OF INSTRUMENT RUN LOG

Katahdin Analytical Services, Inc.
Instrument: GC01
Amount Injected 2 µl
Reviewed by/ Date:

GC Laboratory Instrument Runlog
Method: SW846 (8081) / 8082 / 8151
(circle) EPA 504.1 / 556 / 608
OLC03.2 / SOM01.2
OLM03.2 / OLM04.3

Date	Init.	Result File	Sample ID	Y/N	Method	Column	Comments
042110	RCT	1DD00251	WG76497-3 3550	Y	PEST035	327/328	
		252	WG76282-3 3510				↑methB
		253	-4				
		254	SD1976-1				
		255	-2				
		256	-3				
		257	-4				
		258	SD2108-1 3550				
		259	Hexane	N			
		260	INDAB 0.025	Y			3↑B,2↑A P5755
042210		261	Hexane	N			
		262	eval	Y			P5944
		263	INDAB 0.05	Y			↑methA&B P5754
		264	TC 0.5	Y	PEST035C		
		265	0.05				
		266	0.1				
		267	0.25				
		268	1				
		269	2.5				
		270	WG76373-1 3550	Y			
		271	-2				↓ESIIA, ↓ESI Both
		272	SD1976-5 3510	Y			
		273	-8				
		274	-9				↓DCBA&B
		275	-10				↓DCBA&B
		276	-11				
		277	SD1789-3DL 3550				1:5 100/1000 ↑DCBA, ↓DDD
		278	-8DL				1:2 500/1000 ↑DCBA
		279	SD1837-4RA				
		280	Hexane	N			

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TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8081

FIGURE 2

DATA REVIEW CHECKLIST

PRIMARY REVIEW CHECKLIST

Verbal Due Date _____ **(** ☐ **Verbals turned in). DueDate**_____

Client:		Primary	Secondary
Method:		Date:	Date:
SDG No:	Level:	Initials:	Initials:
KAS No:			Approved : ☐ Yes

DODQSM 3.0 ☐ **DODQSM 4.1** ☐ **WITH LAB. LIMITS** ☐
QUAPP ☐ **LAB** ☐ **(** *REPORT* ***ND's to MDL*** ☐ **)**

List all curves that are scanned. _____ _____

_____ _____ _____ _____

Narrate which QC limits were used for (Surr., LCS,s MS/MSDs.) _____

All needed forms are present . _____

Correct Work Order Number or SDG name (all forms). _____

Correct project name and spelling (all forms). (Truncated ☐). _____

Correct file numbers (all forms). _____

Analysis Date Correct. _____

Extraction Method & Analysis Method Correct. _____

Product list compared to ROAs (compounds & PQLs). _____

Chromatogram reviewed for unlabeled peaks (check product list). _____

Flagging of all ROAs correct (Florida Flagging ☐). _____

All tunes included (level IV) . _____

All log book pages included (Soil weights,TCLP & SPLP). _____

Verify DOD QSM criteria. _____

Narrate any method deviations. (Blanks, LCS,s etc.) _____

Sign & Date Manual integration **(Narrate as needed).** _____

Sample I.D's Truncated (**NARRATED**). YES ☐ Please list KAS # below :

_____

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TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8081

FIGURE 3

PQLS FOR METHOD 8081

Parameter/Method	Analyte	Practical Quantitation Level (PQL)	
		Waters (ug/L)	Soils (ug/kg)
Organochlorine	Aldrin	0.05	1.7
Pesticides	Alpha BHC	0.05	1.7
	Beta BHC	0.05	1.7
SW3510/SW8081B (W)	Delta BHC	0.05	1.7
SW3520/SW8081B (W)	Gamma BHC (Lindane)	0.05	1.7
SW3550/SW8081B (S)	Chlordane	0.50	17
	alpha-Chlordane	0.05	1.7
	gamma-Chlordane	0.05	1.7
	4,4'-DDD	0.10	3.3
	4,4'-DDE	0.10	3.3
	4,4'-DDT	0.10	3.3
	Dieldrin	0.10	3.3
	Endosulfan I	0.05	1.7
	Endosulfan II	0.10	3.3
	Endosulfan Sulfate	0.10	3.3
	Endrin	0.10	3.3
	Endrin Aldehyde	0.10	3.3
	Endrin Ketone	0.10	3.3
	Heptachlor	0.05	1.7
	Heptachlor Epoxide	0.05	1.7
	Methoxychlor	0.50	17
	Toxaphene	1.00	33

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

Prepared By: Peter Lemay Date: 4/98

Approved By:

Group Supervisor: Peter Lemay Date: 1/15/01

Operations Manager: John C. Bunton Date: 1/15/01

QA Officer: Deborah J. Nadeau Date: 1.22.01

General Manager: Duncan C. Kilgore Date: 1/16/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 8082	Format changes, added pollution prevention, minor changes to sections 7, 8 and Table 1	DN	1.22.01	1/22/01
02 8082	Revised sections 7.3.1, 7.4.5 and 7.6.1 to be compliant with South Carolina requirements.	DN	5.23.01	5.23.01
03 8082	Changed to practice of reporting higher value. Other minor changes to sections 7.5.2, 7.7.3 + to Table 2.	DN	5.21.02	5.21.02
04 8082	Revised SOP to indicate Turbochrom is being used as instrument control + data collection software. Included Target-related definitions. Changes to sections 7.7.3, 7.7.4 and 7.8.	MRC	08.20.04	08.20.04
05 8082	Changed 7.5.2 to reflect alternating CV Changed Table 2 Sect. 7.3.1 New Checklist added wording to sect. 8	LAD	020305	020305

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06 8082	Changed PCB1260 to Aroclor 1260. Removed references to 3541. Updated table 2. Added instructions to shake extract before vialing	LAD	04/06	04/06
07	Added waste streams to sect. 1.0. Added ICV to definitions, sect. 5, sect. 7 and Table 1. Added wording regarding 2nd column confirmation criteria and flagging rules to sect. 7.7.4. Added CCV criteria to sect. 7.5.3 and Table 1. Added wording regarding MI to sect. 7.7.3	LAD	08/07	08/07
08	Added tissue, wipe and oil matrices. Added extraction method 3535. Added DDT analog interference, Std. information and analysis frequency criteria. Added HTs are a recommendation. Added note that 2 detectors must be used for dual column. Updated method references Removed calibration and surrogate method mod. from Table 2. Added more info @ linear calib. Added extraction references,	LAD	02/09	02/09
09	Added Chemstation to definitions. Clarified that surrogates are added to only the aroclor 1660 standards, not ALL standards.	LAD	05/09	05/09
10	Revised sections 7, 8, and 10 to reflect compliance with the DoD QSM version 4.1	LAD	08/09	08/09
11	Added Table 2 with DoD QSM Ver. 4.1 QC criteria. Minor changes to Table 1.	LAD	04/10	04/10

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

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TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

1.0 SCOPE AND APPLICATION

This SOP describes all aspects of the analysis of extracts of aqueous, solid, tissue, wipe and oil samples for PCBs by EPA Method 8082A as performed by Katahdin Analytical Services, Inc. including sample analysis, data review, standard preparation and instrument calibration.

It is applicable to the following compounds: Aroclor-1242, Aroclor-1254, Aroclor-1221, Aroclor-1232, Aroclor-1248, Aroclor-1260 and Aroclor-1016. Extracts are analyzed by Gas Chromatography-Electron Capture Detector (GC-ECD)

1.1 Definitions

ANALYTICAL BATCH: 20 or fewer samples which are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods.

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, laboratory reagent grade water is used as a blank matrix; for soil samples, muffled sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

CALIBRATION CHECK: Verification of the ratio of instrument response to analyte amount, a calibration check is done by analyzing for analyte standards in an appropriate solvent. Calibration check solutions are made from a stock solution that is different from the stock used to prepare standards.

CALIBRATION STANDARD (WORKING STANDARD): A solution prepared from the stock standard solution that is used to calibrate the instrument response with respect to analyte concentration.

INDEPENDENT CALIBRATION VERIFICATION STANDARD (ICV): A solution prepared from a stock standard solution independent of the calibration mix that is used to verify the calibration.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent

recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

STANDARD CURVE (CALIBRATION CURVE): A curve that plots concentration of known analyte standard versus the instrument response to the analyte.

STOCK STANDARD SOLUTION: A concentrated solution containing a single certified standard that is a method analyte, or a concentrated solution of a single analyte prepared in the laboratory with an assay reference compound. Stock standard solutions are used to prepare calibration standards.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, Aroclor 1660 standard, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

KATAHDIN INFORMATION MANAGEMENT SYSTEM (KIMS): A complete multi-user system with the capabilities of integrating laboratory instrumentation, generating laboratory worksheets, providing complete Lab Order status and generating reports. KIMS utilizes these features through a database.

PE NELSON TURBOCHROM OR HP CHEMSTATION: data acquisition systems that are used to collect chromatographic data. The systems can also be used to archive raw data files.

TARGET: A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

TARGET DB: An oracle database used to store and organize all Target data files.

QUICKFORMS: A laboratory reporting software for Target and Target DB. The QuickForms report module for Target is preconfigured with generalized forms and US EPA CLP report forms and disk deliverables, which can be customized.

1.2 Responsibilities

1.2.1 This method is restricted to use by, or under the supervision of analysts experienced in the analysis of PCBs by method 8082. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, “Personnel Training & Documentation of Capability,” current revision.

1.2.2 It is the responsibility of all Katahdin technical personnel involved in analysis by method 8082 to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

1.2.3 It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Health and Safety

1.3.1 Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

1.3.2 Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

1.4.1 Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Management Program for further details on pollution prevention techniques.

1.4.2 Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Analytical Environmental Health and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

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1.4.3 Wastes generated during standards preparation are disposed of in the Mixed Flammable Waste (O). After the extracts have been analyzed, the autosampler vials and any expired standard vials or ampules are disposed of in the PCB Vial Waste (H).

2.0 SUMMARY OF METHOD

2.1 Method 8082 provides gas chromatographic conditions for the detection of PPB concentrations of certain PCBs. Prior to the use of this method, appropriate sample extraction techniques must be used. Both neat and diluted organic liquids (Method 3580, waste dilution) may be analyzed by direct injection. A 2 to 5 ul aliquot of sample is injected into a gas chromatograph (GC) using the direct injection technique, and compounds in the GC effluent are detected by an electron capture detector (ECD).

2.2 The sensitivity of Method 8082 usually depends on the concentration of interferences rather than on instrumental limitations. If interferences prevent detection of the analytes, Method 8082 may also be performed on samples that have undergone the following cleanups: Method 3660 - Sulfur Cleanup and Method 3665 - Sulfuric Acid Cleanup.

3.0 INTERFERENCES

Interferences by phthalate esters can pose a problem in PCB determinations when using the electron capture detector. Common flexible plastics contain various amounts of phthalates. Care has to be taken to avoid using any plastic materials during the extraction process. Exhaustive cleanup of reagents and glassware may be required to eliminate background phthalate contamination.

Compounds from the sample matrix to which the detector will respond, such as single-component chlorinated pesticides including the DDT series.

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph

4.1.1 GC Hewlett Packard 5890 series I or II connected to the Turbochrom or HP Chemstation data system, or equivalent.

4.1.2 Columns - Instruments are configured with a pre-column originating from the injection port, which is connected to a deactivated glass Y splitter that connects two different columns to two detectors. The most commonly used columns are:

RTX-35 30M x 0.53 mm ID, RTX-5 30M x 0.53 MM ID, or RTX-1701 30M x 0.53 mm ID. Equivalent columns can be used.

4.1.3 Detectors: Electron capture detectors (ECD). Note: Two detectors must be employed when using dual columns.

4.2 Volumetric flasks, class A: sizes as appropriate with the ground-glass stoppers.

4.3 Syringes: various sizes for preparing standards and injecting samples on the instrument.

4.4 Vials: various sizes and types including crimp tops.

4.5 Balances: Analytical, 0.0001 g

4.6 Refrigerator for storage of extracts and standards.

5.0 REAGENTS

5.1 Solvents

5.1.1 Hexane: pesticide quality or equivalent for diluting samples and standards.

5.2 Standards

5.2.1 Stock standard solutions: Solutions purchased from suppliers like Restek or other acceptable retailers. Expiration dates are one year from date of opening vial or sooner if manufacturers date is less. Upon receipt, all standards are logged into the appropriate logbook with the date of receipt, expiration date, source, lot number, solvent and concentration of compounds. Standard solutions are stored at 4°C in polytetrafluoroethylene (PTFE)-sealed containers in the dark.

5.2.2 Calibration standards: Prepared through the dilution of the stock standards with hexane. Expiration date is 6 months or sooner. Information is documented in standards prep logbook. The concentrations of the working PCB calibration standards are 0.05 ug/ml, 0.10 ug/ml, 0.25 ug/ml, 1.0 ug/ml, 2.5 ug/ml, and 10.0 ug/ml. The Aroclor 1660 standard also contain the surrogates Tetrachloro-m-xylene (TCX) and Decachlorobiphenyl (DCB) at the respective concentrations: 0.001 ug/ml, 0.002 ug/ml, 0.005 ug/ml, 0.020 ug/ml, 0.050 ug/ml, and 0.20 ug/ml.

5.2.3 Independent Calibration Verification standard (ICV): Prepared through the dilution of the stock standards with hexane. Expiration date is 6 months or sooner. Information is documented in standards prep logbook. The concentration of the ICV PCB standard is 1.0 ug/ml.

5.2.4 DDT Analog Standard: Standard containing the DDT series in hexane at 0.05 ug/ml.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Extracts must be stored under refrigeration and analyzed within 40 days of extraction.

Note: The holding time above is a recommendation. PCBs are very stable in a variety of matrices, and holding times under the conditions listed above may be as long as a year.

7.0 PROCEDURES

7.1 Extraction

Refer to the appropriate SOPs for the correct extraction procedure. In general, water samples are extracted using methods 3510, 3520 or 3535 while solid samples use methods 3540, 3545, or 3550. Tissue samples are extracted using method 3545. Wipes and oils are generally extracted using method 3580.

7.2 Instrument conditions

Refer to the instrument logbook for the current column and conditions.

Typical conditions are:

Makeup flow: 60 ml/min Ar/Methane or Nitrogen
Column flow: 6 ml/min
Injector Temp: 200
Detector Temp: 300
Oven Ramp: 160(0) - 5/min - 260(10)
Run time: 30 min
Injection size: 2 ul

7.3 Calibration

7.3.1 The GC system is calibrated using the external standard calibration procedure. Six-point calibration standards of Aroclor 1660 (Aroclor 1016 and Aroclor 1260),

Aroclor 1242, Aroclor 1248 and Aroclor 1254 are prepared along with mid-point calibration standards of Aroclor 1221 and Aroclor 1232. If Aroclor 1221 or Aroclor 1232 are suspected, then a six-point curve of the respective Aroclor will be analyzed prior to the analysis and quantitation of the sample.

Each calibration standard is injected using the technique that is used to introduce the actual samples into the GC. Three to five characteristic peaks from each Aroclor are used to calibrate a curve. The Target system will calculate a peak height for all three to five peaks in each Aroclor. A separate calibration curve for each of the three to five peaks can be prepared in Target using the peak height against the concentration of the standard. A non-linear calibration applying a second order polynomial (quadratic fit) equation is used to prepare the curve. In order to be used for quantitative purposes, the Coefficient of Determination (r^2) must be greater than or equal to 0.990. The quadratic equation is:

$$y = ax^2 + bx + c$$

where: y = Instrument response
b = Slope of the line
x = Concentration of the calibration standard
c = the intercept

7.3.2 A non-linear calibration model may not be allowable for certain states, federal programs, or clients. South Carolina does not allow non-linear calibration work originating in their state. In these cases, a linear calibration model must be used. Each calibration standard is injected using the technique that is used to introduce the actual samples into the GC. Three to five characteristic peaks from each Aroclor are used to calibrate a curve. The Target system will calculate a peak height for all three to five peaks in each Aroclor. A separate calibration curve for each of the three to five peaks can be prepared in Target using the peak height against the concentration of the standard.

7.3.2.1 Linear calibration using the average calibration factor

The calibration factor (CF) is calculated using the following formula:

$$CF = A_s / C_s$$

Where: A_s = Peak area (or height) of the analyte or surrogate.
C_s = Concentration of the analyte or surrogate, in µg/L.

To evaluate the linearity of the initial calibration, calculate the mean CF, the standard deviation (SD), and the RSD.

If the RSD of the calibration factor is less than or equal to 20% over the calibration range, then linearity through the origin may be assumed, and the average calibration or response factor may be used to determine sample concentrations.

7.3.2.2 Linear calibration using a least squares regression

$$y = bx + c$$

where: y = Instrument response
b = Slope of the line
x = Concentration of the calibration standard
c = the intercept

The analyst should not force the line through the origin, but have the intercept calculated from the five data points. In addition, do not include the origin (0,0) as a sixth calibration point. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.995. The ICAL must be successful before any samples or other QC check samples can be analyzed.

7.3.3 The AR1660 calibration curve must be checked initially by analyzing a standard containing the same analytes as the curve but prepared from another source. If the response of the analytes from the independent source varies by more than ± 20%, a new independent source standard must be analyzed or a new calibration curve must be prepared and/or analyzed.

7.3.4 The working calibration curve must be verified prior to sample analysis and every 10 samples thereafter by injecting the mid-point calibration standard. If the response for any analyte varies from the expected response by more than ± 15%, a new calibration curve must be prepared for that analyte. The average result for 5 peak heights of the Aroclors is used for quantitation.

For clients or projects requiring DoD QSM 4.1, the response for any analyte must not vary from the expected response by more than ± 20%, or a new calibration curve must be prepared for that analyte. If the CCV fails the above criteria, reanalyze all samples since the last successful calibration verification. If reanalysis cannot be performed, data must be qualified and explained in the narrative. Additionally, apply a Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.

7.4 Retention time windows

7.4.1 Three injections are made of all the PCBs throughout the course of a 72 hour period.

7.4.2 A major peak from the envelope is chosen and a standard deviation is calculated using the three retention times for that peak.

7.4.3 Plus or minus three times the standard deviation of the retention times for each standard is used to define the retention time window; however, the experience of the analyst should weight heavily in the interpretation of chromatograms. The analyst should use the retention time window, but should primarily rely on pattern recognition.

7.4.4 Retention time windows are calculated for each standard on each GC column at method setup and after major maintenance, including whenever a new GC column is installed. The data is kept on file in the laboratory.

7.4.5 If the calculated retention time window results in a value of 0.03 minutes or less, the laboratory will apply nominal windows. This is done in order to avoid any false negative hits because of the window being to narrow. The windows are: ± 0.07 for all target analytes. By utilizing these windows, a false positive hit may be initially indicated, but an experienced analyst could determine a false positive from scrutinizing the chromatograms. Please note that the use of nominal retention time windows may not be allowable for certain states, federal programs, or clients. South Carolina does not allow the use of nominal limits for compliance work originating in their state. In these cases, a window of ± 0.03 minutes must be used if the established retention time window is less than 0.03 minutes.

7.5 DDT Analog standard; This standard should be analyzed to determine if the commonly found DDT analogs (DDT, DDE, DDD) elute at the same retention times of any of the target PCBs. This standard should be analyzed in conjunction with the retention time window determination.

7.6 Gas chromatographic analysis

7.6.1 Shake samples and let them sit for one minute before vialing for analysis.

7.6.2 All instrument injections are performed using the direct injection technique with an autosampler set for 2-5 ul injection volumes.

7.6.3 Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration as listed in section 7.3 followed by

sample extracts interspersed with mid-concentration calibration standards. Before any samples are analyzed the instrument must be calibrated by analyzing a six-point calibration or a 1.0ppm concentration standard (CV-calibration verification standard) for Aroclor 1660, Aroclor 1242, Aroclor 1248 and Aroclor 1254. If a CV is run, the calculated concentration must not exceed a difference of ± 15%. For clients or projects requiring DoD QSM 4.1, the response for any analyte must not vary from the expected response by more than + 20%, or a new calibration curve must be prepared for that analyte. If the CCV fails the above criteria, reanalyze all samples since the last successful calibration verification. If reanalysis cannot be performed, data must be qualified and explained in the narrative. Additionally, apply a Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification. Each sample analysis must be bracketed with an acceptable initial calibration or an opening CV and an ending CV for each 12-hour shift. The closing CV for Aroclor 1660 is a 0.25ppm concentration standard. All other Aroclors at the closing of the run remain at 1.0ppm concentration. If a second window of samples is run immediately after the closing CVs, the concentration of Aroclor 1660 at the completion of this window would be 1.0ppm. The calibration standard must also be injected at intervals of not less than once every ten samples and at the end of the analysis sequence. If the CV fails, the instrument is checked for any obvious problems and maintenance is performed if deemed necessary. Another CV is analyzed or the instrument is recalibrated and then samples are injected. All samples that were injected after the standard exceeding the criterion must be reinjected to avoid errors in quantitation, if the initial analysis indicated the presence of the specific target analyte that exceeded the criterion.

7.6.3.1 However, if the standard analyzed after a group of samples exhibits a response for an analyte that is above the acceptance limit, i.e. >15%, and the analyte was not detected in the specific samples analyzed during the analytical shift, then the extracts for those samples do not need to be reanalyzed, as the CV standard has demonstrated that the analyte would have been detected were it present. In contrast, if an analyte above the QC limits was detected in a sample extract, then re-injection is necessary to ensure accurate quantitation. If an analyte was not detected in the sample and the standard response is more than 15% below the initial calibration response, then re-injection is necessary to ensure that the detector response has not deteriorated to the point that the analyte would not have been detected even though it was present.

7.6.4 The center of the retention time window for each analyte and surrogate is established by using the absolute retention time for each analyte and surrogate from the daily opening calibration verification or initial calibration.

7.6.5 The identification of PCBs is based on agreement between the retention times of peaks in the sample chromatogram with the retention time windows established through the analysis of standards of the target analytes. An analyte is tentatively identified when a peak from a sample falls within the daily retention time window. Each tentative identification must be confirmed using a second GC column of dissimilar stationary phase or using another technique such as GC/MS. If the retention times of the peaks on both columns fall within the retention time windows on the respective columns, then the target analyte identification has been confirmed.

7.6.5.1 An additional criterion is applied for the identification and quantitation of PCBs. Identification is based on the characteristic fingerprint retention time and shape of the major peaks. Major peaks are defined as those peaks in the Aroclor standard that are at least 25% of the height of the largest Aroclor peak. The sample chromatogram is compared to the individual Aroclor standard chromatograms. Once the Aroclor pattern has been identified, a concentration is then calculated in Target.

7.6.5.2 Three to five Aroclor concentrations are calculated using the peak heights of the three to five characteristic peaks of the Aroclor. These three to five concentrations are then averaged to determine the concentration of that Aroclor.

7.6.6 When samples are analyzed from a source known to contain specific Aroclors, the results from a single-column analysis may be confirmed on the basis of a clearly recognizable Aroclor pattern.

7.6.7 If the response for an analyte exceeds the calibration range of the system, the sample must be diluted and reanalyzed.

7.6.8 If peak detection and identification are prevented due to interferences, the hexane extract may need to undergo a cleanup. The extract may be subjected to a sulfur cleanup (method 3660) and/or a sulfuric acid cleanup (method 3665).

Note: Samples routinely receive a sulfuric acid clean up. However, for samples from a known site with a clean matrix, a sulfuric acid clean up may not be performed. Whenever a sample receives a cleanup, the associated QC must also be subjected to the same cleanup(s) and reanalyzed.

7.6.9 When a GC system is determined to be out of control because either a CV cannot pass or a six point calibration does not meet the correlation coefficient criteria, instrument maintenance is likely necessary. Routine instrument maintenance may involve changing the septum, replacing the liner, clipping the

pre-column, or replacing the column. This information is recorded in the instrument run log (Figure 1). When an instrument requires more severe maintenance like replacing the ECD or an electronic board, this information is written in the instrument maintenance logbook.

7.7 Calculations

7.7.1 The concentration of an analyte is calculated by using the calibrated curve that is prepared in Target. When an analyte is identified, Target displays a concentration after the file is processed through the appropriate calibration method. Aroclor quantitation is accomplished by the method described in section 7.5.4.1.1. However, if a sample contains more than one Aroclor, a peak common to both analytes must not be used to quantitate either compound.

7.7.2 The concentrations from the reports are then incorporated with the extraction data to arrive at a final concentration.

Water: Concentration (ug/L) = (C) (Vt)/ (Vs)

Soil/Sediment: Concentration (mg/kg) = (C) (Vt)/ (Ws) (D)

where, C = concentration calculated by Target in ug/ml
Vt = Volume of total extract including any instrument dilutions
Vs = Volume of sample extracted
Ws = Weight of sample extracted
D = Decimal total solids

7.8 Data Review

7.8.1 Initial Data Review

The initial data review is accomplished by the analyst who ran the samples. This review is of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed. The initial data review is performed on the detailed quantitation reports of the analyzed samples. This data review examines criteria that directly impact whether or not the sample needs to be reanalyzed and/or extracted. These criteria include:

- QC criteria for method blank, LCS, MS/MSD, and calibration – refer to section 8.0.
- Surrogate recovery
- Chromatography: cleanups, manual integration.
- Target compound detection: quantitation, confirmation, false positives.

The requirement of the GC laboratory is that this initial data review be completed no later than the end of the next workday. After the analyst has completed his or her initial data review, the information is then ready to be processed for reporting. Refer to section 7.8.

7.8.2 Surrogate recovery

All recoveries must meet the most recently laboratory established acceptance limits, which are listed on the GC Laboratory Surrogate Acceptance Limit sheet.

The sample is evaluated for recoveries of the two surrogates. If the recovery of one surrogate is within the acceptance limit, and the second is out, the data is narrated. If the surrogate recoveries are high for both and the sample contains less than the PQL for all target analytes, the data is narrated. If the surrogate recoveries are low and may be attributable to matrix interference or a matrix effect, the data is narrated. If the surrogate recoveries are low and the sample concentration is less than the PQL for all target analytes and there is no apparent matrix effect, reextract the sample.

For method blanks, if the recoveries of both surrogates are low or high, and the blank does not contain any target analytes above the PQL, and the recoveries of both surrogates in the sample(s) are acceptable, the data is narrated. If the recoveries in the blank are low and it does not contain any target analytes above the PQL, and the recoveries in the samples are acceptable but the sample contains one or more target analytes above the PQL, the sample may be reextracted.

For laboratory control samples (LCS), if the only discrepancy in the extraction batch is with the LCS, and the analyte spike recoveries are acceptable, the data is narrated. If the recoveries of both the surrogates and the analyte spikes are low, the samples may need to be reextracted.
For DoD QSM 4.1, use QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits. When the surrogate recoveries fall outside of the acceptance criteria, apply Q-flag to all associated analytes.

7.8.3 Chromatography

The chromatography should be examined for the presence of any non-target peaks, which can be used as an indication of whether or not matrix interference might be influencing surrogate recoveries. If the chromatogram indicates interferences, then a cleanup may be needed. See section 7.5.7.

Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating the peak of concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.
Each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. Should a manual integration be necessary (for instance, due to a split peak, peak tailing, or incomplete resolution of isomeric pairs), an "m" qualifier will automatically be printed on the quantitation report summary. The analyst will date and initial the "m" on the quanitation report summary and assign a code that indicates the reason for the manual integration. Refer to Katahdin SOP QA-812 "Manual Integration on GC/MS, GC, HPLC and IC Datasystems" for more information.

7.8.4 Target Compound Detection

GC analysis relies heavily on the experience of the analyst. Sample chromatograms must be evaluated focusing on scientific judgment, knowledge of the column behavior and matrix effects. The chromatogram from channel A is evaluated with that from channel B. If a target analyte is present on both channels and the concentration is within the calibration range, and the quantitation from both chromatograms agrees within ±40%, the analyte is considered present in the sample. In cases where the RPD is greater than 40% and the analyte is reported, the analyte must be J-flagged and narrated. The higher of the two concentrations is reported unless matrix interference is causing erroneously high results. In this case report the lower result and narrate. In some cases a non-confirming analyte may be reported. In these cases the analyte must be Q-flagged and narrated...

In order to avoid reporting false positives, identified peaks on a chromatogram may need to be undetected electronically in Target. The possible scenarios are: If an analyte is present on one column but its concentration is below the PQL, if an analyte is present on one column but does not confirm on the other channel, if an analyte is present on both columns but the concentrations differ by more than 40%, or if an analyte is present but its retention time is ±0.04 minutes or more than the retention time of the analyte in the preceding CV. The GC Analyst must rely on technical experience in reviewing chromatograms in determining if a hit is an actual analyte or a false positive.

If reporting data that has an RPD that is >40%, the data must be flagged with a "J" indicating that the result is an estimated value. Sometimes interference on one column (i.e. sulfur) will prevent a target analyte from detection and it is present on the conformational column. In this scenario, the result would be

reported from one column and need to be "Q" flagged to indicate that it was not confirmed on a second column.

7.8.5 Reporting

After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into QuickForms. Depending on the QC level requested by the client, a Report of Analysis (ROA) and additional reports, such as LCS forms and chronology forms, are generated. The package is assembled to include the necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded to the secondary reviewer. The secondary reviewer validates the data and checks the package for any errors. When completed, the package is sent to the Department Manager for final review. A completed review checklist is provided with each package. The final data package from the Organics department is then processed by the Data Management department.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 and to details in this section for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The criteria does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in this section or in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in this section and in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

8.1 For each analytical batch (up to 20 samples), a method blank, laboratory control sample (LCS), matrix spike and matrix spike duplicate are analyzed. They are carried through all stages of the sample preparation and analysis steps.

8.2 Spike concentrations: The LCS and the MS/MSD are spiked at the same concentration with Aroclor 1660. The spike concentrations are:

Compound	WATER ug/L	SOILS mg/kg
Aroclor 1660	5.0	0.17

The surrogate spike concentrations in the final extract are:

Compound	WATER ug/ml	SOILS ug/ml
Tetrachloro-m-xylene(TCX)	0.10	0.10
DCB	0.10	0.10

8.3 LCS and MS/MSD acceptance criteria and Corrective Action: All QC samples are calculated for percent recovery of the spiked analyte. The recoveries are compared to laboratory established acceptance limits. The LCS acceptance limits for PCBs are established for both water and soil matrices. The MS/MSD acceptance limits for PCBs use the respective matrix LCS acceptance limits. Separate limits for MS/MSD pairs are not calculated because of the varying matrices involved. In addition many of the MS/MSD data points cannot be used (i.e. recoveries not calculable due to a matrix effect).

If any spike compound in the laboratory control sample falls outside of the established recovery acceptance limit window, the QC sample is considered to be out of control and any sample that is associated should be evaluated with other QC elements to determine the corrective action. If the recovery is high and the associated samples do not contain the specific compound(s), the data can possibly be accepted with narration. In other cases, the associated samples must be extracted.
If a spike compound is outside of the acceptance limits in the matrix spike sample but is acceptable in the LCS, the data is considered acceptable. The cause of the failure is possibly attributable to matrix interference. However, if the compound fails in both the LCS and the MS/MSD, the result for that analyte is suspect and may not be reported for regulatory compliance purposes.

For DoD QSM 4.1, use QC acceptance criteria specified by DoD, if available. Otherwise use in-house control limits. In-house control limits must not be greater than $\pm$ 3 times the standard deviation of the mean LCS recovery. If the LCS fails the acceptance criteria, correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. If reanalysis cannot be performed, data must be qualified and explained in

the narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.

For MS, when applying DoD QSM 4.1, apply J-flag to specific analyte(s) also in parent sample, if acceptance criteria not met. RPD must be $\leq$ 30% between MS and MSD.

8.4 Surrogate acceptance criteria and Corrective Action: Surrogate recoveries are calculated on all samples, blanks and spikes. The recoveries are compared to laboratory established acceptance limits.

When a sample has a surrogate that falls outside of the laboratory established acceptance limit window, the problem should be investigated. If the recovery looks like it is affected by the sample matrix, the sample may be reinjected to confirm matrix interference. When a sample has no detectable surrogate recovery, the sample should be reextracted.

For DoD QSM 4.1, use QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits. When the surrogate recoveries fall outside of the acceptance criteria, apply Q-flag to all associated analytes.

8.5 CAR: Whenever data is not acceptable because of a failing LCS or surrogate recovery, a corrective action report (CAR) must be initiated as soon as possible to document resolution.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined prior to sample analysis per type of instrument and filed with the Organic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revision of Method 8082 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition. Final Update IV, dated February, 2007, Method 8082A.

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

Department of Defense Quality Systems Manual for Environmental Laboratories (DoD QSM), Version 4.1, 04/22/09.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

Katahdin Analytical Services, Inc., SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin Analytical Services, Inc., SOP CA-106, Standard Preparation, Documentation and Traceability, current revision.

Katahdin Analytical Services, Inc., SOP CA-500, Preparation of Soil/Sediment Samples by Sonication Using Method 3550 for Subsequent Pesticides/PCBs Analysis, current revision.

Katahdin Analytical Services, Inc., SOP CA-515, Preparation of Aqueous Samples for Pesticides/PCBs Analysis-Methods 3510 and 3520, current revision.

Katahdin Analytical Services, Inc., SOP CA-524, Preparation of Soil/Sediment Samples by Soxhlet Extraction Using Method 3540 for Subsequent Pesticides/PCBs Analysis, current revision.

Katahdin Analytical Services, Inc., SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

LIST OF FIGURES

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

TABLE 1

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
6pt calibration of Aroclor 1660, 1242, 1248, 1254 and mid-point cal of other Aroclors	Initial cal prior to sample analysis	6 pt calibration – Coefficient of Determination (r^2) ≥ 0.990	(1) Repeat Initial calibration (2) If single pt cal Aroclor is identified in analysis of sample, 6-pt calibration run of identified compound with reanalysis of sample.
5pt calibration of Aroclor 1660, 1242, 1248, 1254 and mid-point cal of other Aroclors	Initial cal prior to sample analysis	5 pt calibration – (r) ≥ 0.990	(1) Repeat Initial calibration (2) If single pt cal Aroclor is identified in analysis of sample, 6-pt calibration run of identified compound with reanalysis of sample.
Independent Calibration Verification	Immediately following calibration	± 20 % D	(1) Reanalyze standard (2) Reprep standard (3) Reprep standard from fresh stock.
CCV	After every 10 samples; If calibration curve previously analyzed, analyze daily before samples.	± 20 % D	(1) Evaluate the samples: If the %D >+15% and sample results are <PQL, narrate. (2) If %D >±15% only on one channel, narrate. If %D >±15% for closing CV, and is likely a result of matrix interference, narrate. (3) Otherwise, reanalyze all samples back to last acceptable CV.
Method blank	One per prep batch	No analyte detected >PQL	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e. If the blank results are above the PQL, report sample results which are <PQL or > 10X the blank concentration. (3) Otherwise, reprep a blank and the remaining samples.
LCS	One per prep batch of twenty or fewer samples	± 15% D	(1) Evaluate the samples and associated QC: i.e. If an MS/MSD was performed and acceptable, narrate. (2) If an LCS/LCSD was performed and only one of the set was unacceptable, narrate. (3) If the surrogate recoveries in the LCS are also low but are acceptable in the blank and samples, narrate. (4) If the LCS recovery is high but the sample results are <PQL, narrate. (5) Otherwise, reprep a blank, QC and the remaining samples.
Matrix Spike\ Matrix Spike Duplicate	One for every set of 20 samples	Same as for LCS	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS/MSD are unacceptable reprep samples and QC.

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

TABLE 1 (cont.)

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Sample Duplicate	One sample duplicate per ten samples if requested	RPD $\leq$20	(1) If lab QC in criteria and matrix interference suspected, flag data or narrate (2) Otherwise, reanalyze
Demonstration of analyst proficiency – 4 replicates	Once per analyst initially and annually thereafter	P&A meet method criteria	(1) Repeat P&A study
MDL study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

TABLE 2

DOD QSM VERSION 4.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria.	Not Applicable (NA).	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification	Refer to current revision of SOP QA-806				
LOQ establishment and verification	Refer to current revision of SOP QA-806				
Retention time (RT) window width calculated for each analyte and surrogate	At method set-up and after major maintenance (e.g., column change).	RT width is ± 3 times standard deviation for each analyte RT from a 72-hour study.	NA.	NA.	
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	6 point calibration of Aroclors 1660, 1242, 1248, and 1254 - One of the options below: Option 1: RSD for each analyte ≤ 20%; Option 2: linear least squares regression: r ≥ 0.995; Option 3: non-linear regression: coefficient of determination (COD) r2 ≥ 0.99 (6 points shall be used for second order). Mid point calibration of Aroclors 1221 and 1232; if targets are detected, 6-point calibration is performed.	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. Calibration may not be forced through the origin. Quantitation for multicomponent analytes such as chlordane, toxaphene, and Aroclors must be performed using a 5-point calibration. Results may not be quantitated using a single point.
Retention time window position establishment for each analyte and surrogate	Once per ICAL and at the beginning of the analytical shift.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

TABLE 2 (cont)

DOD QSM VERSION 4.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Second source calibration verification (ICV)	Immediately following ICAL.	All project analytes within established retention time windows. GC methods: All project analytes within ± 20% of expected value from the ICAL.	Correct problem, rerun ICV. If that fails, repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
calibration verification (CCV)	Prior to sample analysis, after every 10 field samples, and at the end of the analysis sequence.	All project analytes within established retention time windows. GC methods: All project analytes within ± 20% of expected value from the ICAL.	Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed. Retention time windows are updated per the method.
Method blank	One per preparatory batch.	No analytes detected > ½ RL (> RL for common lab contaminants) and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Reprepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result. Contact Client if samples cannot be reprepped within hold time.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory control sample (LCS) containing all analytes to be reported, including surrogates	One per preparatory batch.	The laboratory shall use laboratory control limits (CLs) or use DoD-generated LCS-CLs, if available depending on project requirements. In-house CLs may not be greater than ± 3 times the standard deviation of the mean LCS recovery. A number of analytes may fall outside the CL but within marginal exceedance limit depending on the total number of analytes in the LCS.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. Refer to Table G-1 for number of marginal exceedences allowed. Contact Client if samples cannot be reprepped within hold time.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

TABLE 2 (cont.)

DOD QSM VERSION 4.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix spike (MS)	One per preparatory batch per matrix if sufficient sample is available.	For matrix evaluation, use LCS acceptance criteria.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix Spike duplicate (MSD)	One per preparatory batch per matrix if sufficient sample is available.	MSD: For matrix evaluation, use LCS acceptance criteria. MS/MSD: RPD ≤ 30%.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Surrogate spike	All field and QC samples.	The laboratory shall use laboratory control limits (CLs) or use DoD-generated LCS-CLs, if available depending on project requirements.	For QC and field samples, correct problem then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary. Contact Client if samples cannot be reprepped within hold time.	Apply Q-flag to all associated analytes if acceptance criteria are not met.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Confirmation of positive results (second column or second detector)	All positive results must be confirmed (with the exception of Method 8015).	Calibration and QC criteria same as for initial or primary column analysis. Results between primary and second column RPD ≤ 40%.	NA.	Apply J-flag if RPD > 40%. Discuss in the case narrative.	Use project-specific reporting requirements if available; otherwise, use method reporting requirements; otherwise, report the result from the primary column (see Box D-16).
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

TABLE 3

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-329-11	METHOD 8082, current revision
Procedures	7.4.5 If the calculated retention time window results in a value of 0.03 minutes or less, the laboratory will apply nominal windows. This is done in order to avoid any false negative hits because of the window being to narrow. The windows are: ± 0.07 for all target analytes. By utilizing these windows, a false positive hit may be initially indicated, but an experienced analyst could determine a false positive from scrutinizing the chromatograms. Please note that the use of nominal retention time windows may not be allowable for certain states, federal programs, or clients. South Carolina does not allow the use of nominal limits for compliance work originating in their state. In these cases, a window of ± 0.03 minutes must be used if the established retention time window is less than 0.03 minutes.	9.3 refers to method 8000B section 7.6.3: If the standard deviation of the retention times for a target compound is 0.000 (i.e., no difference between the absolute retention times), then the laboratory may either collect data from additional injections of standards or use a default standard deviation of 0.01 minutes. (Recording retention times to three decimal places rather than only two should minimize the instances in which the standard deviation is calculated as 0.000).
Apparatus/Materials		
Reagents		
Sample Preservation and handling		
QC – Spikes		
QC – LCS		
QC – Accuracy/ Precision		
QC - MDL	PQL Practical Quantitation Level – three to ten times the MDL.	EQL Estimated Quantitation Level – five to ten times the MDL

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

FIGURE 1

EXAMPLE OF INSTRUMENT RUN LOG

Katahdin Analytical Services, Inc.
GC Laboratory Instrument Runlog
Instrument: GC06
Amount Injected 2 µL

Method: SW846 8082 or EPA 608
Reviewed by/ Date:

Date	Init.	Result File	Sample ID	Y/N	Method	Column	Comments
7.31.07	SJC	GAG3/4152	WG41666-3 3510	Y	PCB/8074A	298/299	
		153	Hexane	N			
		154	AR1660 0.25	Y			↑DC6 ↑1260 both P4274
		155	AR1242 1.0	Y			P4274
		156	AR1248				P4275
		157	AR1254				P4276
		158	SA3963-1 3540				
		159	SA3987-1				
		160	-2				
		161	-3				
		162	-4				
		163	SA3978-1				
		164	-2				
		165	-3				
8.1.07		166	-4				
		167	-5	N			needs SC.
		168	Hexane				
		169	AR1660 1.0	Y			P4273
		170	AR1242				P4274
		171	AR1248				P4275
		172	AR1254				P4276
		173	SA3978-6 3540	N			needs SC
		174	SA3958-4 3510	Y			
		175	-6				
		176	WG41667-1				
		177	-2				
		178	-3				
		179	-4				
		180	~~SA3958-5~~				
		181	SA3958-1				
		182	-2				
		183	Hexane	N			

QAGC167 0000085

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

FIGURE 2

DATA REVIEW CHECKLIST

PRIMARY REVIEW CHECKLIST

Verbal Due Date _____ **(** ☐ **Verbals turned in). DueDate**_____

Client:		Primary	Secondary
Method:		Date:	Date:
SDG No:	Level:	Initials:	Initials:
KAS No:			Approved : ☐ Yes

DODQSM 3.0 ☐ **DODQSM 4.1** ☐ **WITH LAB. LIMITS** ☐
QUAPP ☐ **LAB** ☐ **(** *REPORT* ***ND's to MDL*** ☐ **)**

List all curves that are scanned. _____ _____

_____ _____ _____ _____

Narrate which QC limits were used for (Surr., LCS,s MS/MSDs.) _____

All needed forms are present . _____

Correct Work Order Number or SDG name (all forms). _____

Correct project name and spelling (all forms). (Truncated ☐). _____

Correct file numbers (all forms). _____

Analysis Date Correct. _____

Extraction Method & Analysis Method Correct. _____

Product list compared to ROAs (compounds & PQLs). _____

Chromatogram reviewed for unlabeled peaks (check product list). _____

Flagging of all ROAs correct (Florida Flagging ☐). _____

All tunes included (level IV) . _____

All log book pages included (Soil weights,TCLP & SPLP). _____

Verify DOD QSM criteria. _____

Narrate any method deviations. (Blanks, LCS,s etc.) _____

Sign & Date Manual integration **(Narrate as needed).** _____

Sample I.D's Truncated (**NARRATED**). YES ☐ Please list KAS # below :

_____

QA-044 – Revision 1 – 04/05/2010

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

FIGURE 3

PQLs FOR METHOD 8082

ANALYTE	Practical Quantitation Level (PQL) (ug/L)	Practical Quantitation Level (PQL) (ug/kg)
PCB-1016	0.50	17
PCB-1221	0.50	17
PCB-1232	0.50	17
PCB-1242	0.50	17
PCB-1248	0.50	17
PCB-1254	0.50	17
PCB-1260	0.50	17

TITLE: DETERMINATION OF PETROLEUM RANGE ORGANICS (PRO) BY FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION METHOD # FL-PRO

Prepared By: Peter Lemay Date: 7/18/01

Approved By:

Group Supervisor: Peter Lemay Date: 7/18/01

Operations Manager: Dennis Neiman by John Burdick Date: 7/18/01

QA Officer: Deborah J. Nadeau Date: 7/18/01

General Manager: Dennis F. Neiman Date: 7/18/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Added definitions and information for new data processing system. Added or changed wording to clarify sections 6 and 7 and Table 2. Added wording to sections 8 and 9 per recent NELAC & Navy audits. Minor changes throughout. New figures 1 and 2.	HRC	11.15.04	11.15.04
02	Changed LIMS to KIMS Sodium Sulfate is purified at vendor added wording to sect. 7.7.2 to clarify	LAD	03/06	03/06
03	Many changes made throughout, including but not limited to, waste management, CV frequency, Spike amounts, statistically derived QC limits and method modifications. Please refer to the QAM/SOP change form filed with the SOP in QA for a detailed list of changes	LAD	09/07	09/07
04	Removed Apparatus and Reagents that are not used. Updated surrogate information	LAD	09/08	09/08

TITLE: **DETERMINATION OF PETROLEUM RANGE ORGANICS (PRO) BY FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION METHOD # FL-PRO**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ____ of document **SOP CA-333-04**, titled **DETERMINATION OF PETROLEUM RANGE ORGANICS (PRO) BY DEPARTMENT OF ENVIRONMENTAL PROTECTION METHOD # FL-PRO**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ____ of document **SOP CA-333-04**, titled **DETERMINATION OF PETROLEUM RANGE ORGANICS (PRO) BY FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION METHOD # FL-PRO**

Recipient: _____ Date: _____

TITLE: **DETERMINATION OF PETROLEUM RANGE ORGANICS (PRO) BY FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION METHOD # FL-PRO**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the method used by Katahdin Analytical Services technical personnel to measure the concentration of petroleum range organics (PRO) in water and soil. These compounds correspond to a hydrocarbon range of C_8-C_{40}.

This method is based on a solvent extraction, Gas Chromatography (GC) procedure. The method is designed to measure the petroleum concentration in environmental samples in the above stated C-Range (nominally diesel through motor oils). It cannot be used as an indication of gasoline contamination. Additional analyses may be performed including, but not limited to, analysis of additional reference materials. These additional efforts are not contained within this method.

1.1 Definitions

PETROLEUM HYDROCARBONS: All chromatographic peaks, both resolved and unresolved, eluting between the peak start of n-octane (n-C_8) and the peak end after n-tetracontane (n-C_{40}). Quantitation is based on direct comparison of the area within this range to the total area of the Petroleum Hydrocarbon standard as determined from FID response using baseline-baseline integration.

PETROLEUM HYDROCARBON STANDARD: A 17-component mix of all even numbered normal alkanes from C8 to C40. This standard serves as a quantitation standard and a retention time window defining Petroleum Hydrocarbons.

SAMPLE MATRIX SPIKE: A selected sample from the analytical batch spiked with the Petroleum Hydrocarbon Standard and surrogate standards. The calculated spike recovery shall be used as a control.

LABORATORY CONTROL SAMPLE: Laboratory reagent grade water or standard soil spiked with the Petroleum Hydrocarbon standard and surrogate standards. The calculated spike recovery may be used as a laboratory control.

METHOD DETECTION LIMIT (MDL): Minimum concentration that an analyte can be measured and reported with a 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. The MDL is determined using EPA Appendix B to Part 136, CFR 40 Ch. 1(7-1-94) using the Student t Test.

KATAHDIN INFORMATION MANAGEMENT SYSTEM (KIMS): A complete multi-user system with the capabilities of integrating laboratory instrumentation, generating laboratory worksheets, providing complete Lab Order status and generating reports. LIMS utilizes these features through a database.

PE NELSON TURBOCHROM: A data acquisition system that is used to collect chromatographic data. The system can also be used to archive raw data files.

TARGET: A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

TARGET DB: An oracle database used to store and organize all Target data files.

QUICKFORMS: A laboratory reporting software for Target and Target DB. The QuickForms report module for Target is preconfigured with generalized forms and US EPA CLP report forms and disk deliverables, which can be customized.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of PRO by Method FL-PRO. Analysts should be skilled in the interpretation of gas chromatograms and their use as a quantitative tool. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in analysis of PRO by Method FL-PRO to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures and the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of a respirator and all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the preparation of standards etc. should be disposed of in the "D" waste stream satellite accumulation area nearest the point of generation. Acetone and hexane are considered flammable waste, and should be disposed of in the "O" waste stream satellite accumulation area nearest the point of generation. FLPRO sample vials are considered "P" waste and should be disposed of in the corresponding satellite waste accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

2.0 SUMMARY OF METHOD

2.1 One liter of water or a specified quantity of soil (extraction method dependent) is spiked with two surrogates and extracted with Methylene chloride. The water is removed from the extract, concentrated to a volume of 2.0 mL, and treated with silica gel to remove potential organic interferences. An aliquot is injected onto a capillary column gas chromatograph (GC) equipped with a flame ionization detector (FID). Quanitation is based on the detector response compared to a series of normal alkane standards. This method is suitable for the analysis of waters, soils or wastes.

2.2 This method is based in part on USEPA Methods 8000 and 8100, SW-846, "Test Methods for Evaluating Solid Waste", 3rd Edition, Method OA-2, work by the EPA UST Work Group "Measurement of Petroleum Hydrocarbons: Report on activities to Develop a Manual", 1990, Method AK103.0, Revision 2, PUBL-SW-141, July 1993 and the Florida Department of Environmental Protection Technical Advisory Committee for 62-770, F. A. C, Petroleum Contamination Site Cleanup Criteria.

3.0 INTERFERENCES

3.1 Other organic compounds including chlorinated hydrocarbons, phenols, and phthalate esters are measurable. As defined in the method, the PRO results include these compounds. Spills of known specific constituents should be analyzed and quantified by a method specific for those compounds.

3.2 Method interferences are reduced by washing all glassware with hot soapy water and then rinsing it sequentially with tap water, methanol or acetone, and Methylene chloride. Method blanks must be analyzed with each batch to demonstrate that the samples are free from method interferences.

3.3 High purity reagents (pesticide grade or better) must be used to minimize interferences.

3.4 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. Whenever an unusually concentrated sample is encountered, it should be followed by analysis of a solvent blank to check for cross-contamination.

3.5 Animal and vegetable oil and grease and biogenic terpenes are also measurable if the sample is not cleaned up before analysis. In order to eliminate false positives from these sources, the silica cleanup is a mandatory part of the procedure.

4.0 APPARATUS AND MATERIALS

4.1 Gas Chromatograph: Analytical system complete with gas chromatograph and all required accessories, including a detector, column supplies, recorder, gases, and syringes. A capillary split/splitless injector operating in the splitless mode is recommended. A data system capable of determining peak areas by integrating from baseline to baseline is required.

4.2 Column 1: 30 m x 0.53 mm ID ZB-5, 1.5 micron film thickness (or equivalent). Column 2: 30 m x 0.53 mm ID ZB-1, 1.5 micron film thickness (or equivalent). The

column must be capable of resolving typical diesel components, and the solvent front from C_8. Other columns may be used if all column performance criteria are met.

4.3 Detector: Flame ionization detector (FID).

4.4 Microsyringes: 1 ul, 5 ul, 10 ul, 25 ul, and 100 ul.

4.5 Disposable pipettes: Pasteur.

4.6 2 ml (and larger) vials with Teflon lined caps for storage of extracts.

5.0 REAGENTS

5.1 Solvents: Methylene chloride: Pesticide grade or equivalent. Store away from other solvents.

5.2 Stock Standards: Aliphatic Hydrocarbon standard mix from a vendor like UltraScientific at a concentration of 500 ug/mL in hexane (each of the 17 components from C_8 to C_{40}). A surrogate solution containing n-Triacontane-d_{62} at a concentration of 5000 ug/mL and another surrogate solution containing o-Terphenyl at a concentration of 2000 ug/mL from a vendor like Restek.

5.3 Calibration Standards: The standards are prepared at the following five different concentrations: 200 ug/ml, 100 ug/ml, 50 ug/ml, 20 ug/ml, and 5 ug/ml (per each component). This is equivalent to 85, 340, 850, 1700, and 3400 ug/ml total alkanes in the standards. The concentration of OTP and triacontane-d_{62} must remain at a constant 50 and 300 ug/ml level in all concentration levels.

5.3.1 Transfer the stock standard solution into a Teflon-sealed screw-cap/crimp cap bottle. Store, with minimal headspace, at 6°C or less and protect from light.

5.3.2 Working standards must be replaced after 6 months, or sooner if comparison with check standards indicates a problem.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

6.1 Whenever possible, samples should be grab samples which are collected directly into the sample container. Sample collection equipment such as bailer or intermediate containers should be avoided (exceptions: collection from monitoring

wells or grab samples in surface water at depth). Unless required by permit, automatic samplers may not be used. Pumps such as bladder pumps or peristaltic pumps shall not be used.

6.2 All sampling equipment which contacts the sample shall be constructed of teflon®, stainless steel or glass. Under no circumstances can flexible PVC tubing, such as tygon®, be used in the purging or sample collection process.

6.3 Water samples shall be collected in a one liter glass container; soils in a glass jar. All containers shall be sealed with a screw cap with teflon® liner. Water samples shall be acidified to a pH of less than 2 with hydrochloric or sulfuric acid (reagent grade or better).

6.4 The samples shall be stored at 4°C (±2°C) from the time of collection until extraction. Extraction shall be performed on waters within seven days of sample collection and on soils within 14 days of sample collection. All analyses must take place within 40 days of extraction.

7.0 PROCEDURES

7.1 Waters are extracted using a separatory funnel or continuous liquid liquid extraction technique. Soils are extracted using a sonication technique. Alternatively, soils may be extracted by a Soxhlet extraction technique. Refer to Katahdin SOP CA-520, current revision, for sample preparation procedures. After the extracts are concentrated, an appropriate volume (usually 1ul) is injected directly into the GC. (Recommend using splitless injection techniques).

NOTE: NaCl may be added to water samples to improve extraction efficiency.

If the sample concentration exceeds the calibration range for PRO an appropriate dilution should be used. An appropriate dilution is one that keeps the response of major constituents (previously saturated peaks) in the linear range of the detector. If an initial dilution does not accomplish this then an intermediate dilution should be performed.

7.2 Gas Chromatography:

7.2.1 Conditions (For both column 1 and 2): Set column temperature to 60°C for 2 minutes, then 10°C/min. to 300°C and hold for 24 min. Set FID Detector to 310°C and injector to 300°C. Conditions may be altered to improve resolution or recovery of petroleum range organics.

7.2.2 Performance Criteria: GC run conditions and columns must be chosen to meet the following criteria:

7.2.2.1 Resolution of C_8 from the solvent front.

7.2.2.2 The column must be capable of separating typical petroleum hydrocarbon components from the surrogates.

7.3 Retention Time Window

7.3.1 Before establishing windows, be certain that the GC system is within optimum operating conditions. Make three injections of the method standard throughout the course of a 72 hour period. Serial injections over less than a 72 hour period result in retention time windows that are too tight.

7.3.2 Calculate the standard deviation of the absolute retention times for the two surrogates, C_8, and C_{40}.

7.3.2.1 The retention time window for individual peaks is defined as a plus or minus three times the standard deviation of the absolute retention time for each component.

7.3.2.2 In those cases where the standard deviation for a particular analyte is zero, the laboratory should use ±0.05 min as a retention time window.

7.3.3 The laboratory must calculate retention time windows for these standards on each GC column and whenever a new GC column is installed. The data are retained by the laboratory.

7.4 PRO Calibration

7.4.1 Initial Calibration – Calibration shall be by external calibration using a minimum of 5 concentration levels for the initial calibration. Quantitation shall be by linear regression.

In all cases, response of the standards must be determined by continuous integration of all responses (excluding surrogates) from a forced baseline beginning at a point prior to the elution of C_8 to a point past C_{40}. All responses must be determined as responses to baseline and not valley to valley. A method is calibrated for all five levels using the area of each of the 17 individual alkanes and the area of the two surrogates and a total area of the Petroleum Hydrocarbon Standard (PRO) (which is the total area of the seventeen alkanes for each level).

7.4.1.1 Linear Regression – The linear regression shall be calculated using the total PRO area versus the PRO concentration. The correlation coefficient shall be equal to or greater than 0.995.

7.4.1.2 The accuracy of the initial calibration shall be verified by injecting a midpoint concentration of a standard mix that has been obtained from a different source. The calculated value shall be within ± 20% of the expected value.

7.4.2 Continuing Calibration – The calibration curve must be verified on each working day by the injection of a continuing calibration standard (CV) at a midpoint concentration. This standard must be evaluated prior to the analysis of samples.

In addition, a continuing calibration must be run every 10 samples and at the end of the sequence. The concentration of these should vary, with at least one at a level of 1-2 times the calculated PQL as a verification of sensitivity. To accomplish this, continuing calibrations at 50 ug/ml and 20 ug/ml (each component) should be ran.

7.4.2.1 If the concentration of this standard varies from the predicted concentration by more than ± 25%, a new initial calibration curve must be prepared and verified before samples are analyzed.

7.4.2.2 Retention Time Window – Establish daily retention time windows for each analyte of interest using the absolute retention time for each analyte as the midpoint of the window for that day **if** after analyzing the midpoint it is determined that one or more analytes falls outside of the previously established absolute retention time window. The daily retention time window equals the midpoint ± three times the standard deviation determined in Section 7.3.

7.5 Gas Chromatography Analysis

7.5.1 A 1 ul injection volume is analyzed by GC/FID.

7.5.2 If an initial calibration has already been performed, verify the calibration by analysis and evaluation of a mid-point CV on each working day.

In addition, a CV must be run every 10 samples and at the end of the sequence.

7.5.3 Evaluate the CV per 7.4.2.1 and 7.4.2.2. If either performance criteria fails, the instrument must be recalibrated and all samples which were injected after the failed standard must be reanalyzed.

7.5.4 A Methylene chloride blank will be run in every sequence to determine the area generated on normal baseline bleed under the conditions prevailing in the 24 hour period if requested by the client. This area is determined by continuous integration of all responses under the same conditions (i.e. forced baseline and predetermined time interval) as the samples. This blank is calculated as the solvent blank and the value should be less than the PQL.

Methylene chloride blanks should also be run after samples suspected of being highly concentrated to prevent carryover. If the blank analysis shows contamination, additional blanks should be analyzed until the system is shown to be free from contaminants.

7.5.5 If the sample concentration exceeds the linear range of the method in the final extract, the extract must be diluted and reanalyzed.

7.5.6 Baseline correction is allowed to correct for rises due to temperature programming. Range integration is corrected by the automatic subtraction of the baseline established by activation of a programmed run without the injection of any material. Instrument baseline must be established for every batch of samples.

7.6 Calculations

7.6.1 The integrated area for all peaks eluting from n-octane through n-tetracontane shall be determined using a baseline drawn from the baseline point to n-octane to a point past n-tetracontane where the baseline returns to normal. All area including the "hump-a-gram" and surrogate standards shall be included. Do not integrate valley to valley for individual peaks except for the two surrogates. The concentration of the PRO is calculated by using the calibrated curve that is prepared in Target. Target displays a concentration when the file is processed through the appropriate calibrated method.

7.6.2 The concentrations from the reports are then incorporated with the extraction data to arrive at a final concentration.

7.6.2.1 Water: Conc (ug/L) = (Amt) (DF) ((Vt/Vo) 1000)

7.6.2.2 Soil/Sediment: Conc (mg/kg) = (Amt) (DF) ((Vt/Vo) (100/(100-M)))

where,

Amt = adjusted concentration calculated by Target in ug/ml
Vt = Volume of total extract
Vo = Volume or weight of sample extracted
M = % Moisture
DF = Dilution Factor

7.7 Data Review

7.7.1 Initial Data Review

The initial data review is accomplished by the analyst who ran the samples. This review is of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed. The initial data review is performed in Target Review. This data review examines criteria that directly impact whether or not the sample needs to be reanalyzed and/or extracted. These criteria include:

- QC criteria for method blank, LCS, MS/MSD, and calibration – refer to section 8.0.
- Surrogate recovery
- Chromatography: manual integration.
- Target compound detection: quantitation, false positives.

The requirement of the GC laboratory is that this initial data review be completed no later than the end of the next work day. After the analyst has completed his or her initial data review, the information is then ready to be processed for reporting. Refer to section 7.8.

7.7.2 Surrogate recovery

The recoveries for o-Terphenyl are compared to the method acceptance limits. The recoveries for n-Triacontane-d_{62} are compared to nominal acceptance limits of 70-130% until laboratory acceptance limits can be established.

The sample is evaluated for recoveries of the surrogate OTP and n-Triacontane-d_{62}. If the recovery is low and there is no apparent matrix effect, the sample should be reanalyzed. If the reanalysis is still low, re-extract. If the recovery is low and there may be a matrix effect, reanalyze to confirm a matrix effect and narrate. If the surrogate is high and the sample results are less than the PQL, or there is likely a matrix effect, narrate.

7.7.3 Chromatography

The chromatography should be examined for the presence of any non-target peaks, which can be used as an indication of whether or not matrix interference might be influencing surrogate recoveries.

Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating the peak of concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.

In Target Review, each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. Should a manual integration be necessary (for instance, if the sample contains a concentration of PRO which was integrated "valley to valley" instead of a "baseline to baseline"), manual integration is performed in Target Review. A "m" qualifier will automatically be printed on the quantitation report summary indicating that a manual integration was performed. For specific procedures on how to manually integrate, refer to Katahdin SOP QA-811, Manual Integration, current revision.

7.8 Reporting

After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into QuickForms. Depending on the QC level requested by the client, a Report of Analysis (ROA) and additional reports, such as LCS forms and chronology forms, are generated. The package is assembled to include the necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded to the secondary reviewer. The secondary reviewer validates the data and checks the package for any errors. When completed, the package is sent to the Department Manager for final review. A completed review checklist (Figure 2) is provided with each package. The final data package from the Organics department is then processed by the Data Management department.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

See below or refer to Table 1 for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective

actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The Department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. It may not be possible to reanalyze samples within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 For each analytical batch (up to 20 samples), a method blank, laboratory control sample (LCS), matrix spike and matrix spike duplicate or sample duplicate are analyzed. They are carried through all stages of the sample preparation and analysis steps.

8.2 The laboratory shall generate control limits based on +/-3 standard deviations from the average recovery for all spikes and surrogates, and + 3 standard deviations from the average precision value for all duplicates. The limits that area generated must be within the criteria specified in Table 3 below.

8.3 Spike concentrations: The LCS and the MS/MSD are spiked with the seventeen component PRO mix at the same concentration. The spike concentrations are:

	WATER	SOILS
	ug/L	mg/Kg
PRO	850	28.5

The surrogate spike concentrations in the final extract are:

	WATER	SOILS
	ug/ml	mg/kg
o-Terphenyl	50	1.65
n-Triacontane-d_{62}	300	10

8.4 LCS and MS/MSD acceptance criteria and Corrective Action: All QC samples are calculated for percent recovery of the spiked analyte(s). The recoveries are compared to laboratory established acceptance limits.

If any spike compound in the laboratory control sample falls outside of the established recovery acceptance limit window, the QC sample is considered to be out of control and any sample that is associated should be reextracted. However, if the recovery is high and the associated samples do not contain the specific compound(s), the data can possibly be accepted with narration.

If a spike compound is outside of the acceptance limits in the matrix spike sample but is acceptable in the LCS, the data is considered acceptable. The cause of the failure is possibly attributable to matrix interference. However, if the compound fails in both the LCS and the MS/MSD, the result for that analyte is suspect and may not be reported for regulatory compliance purposes.

8.5 Surrogate acceptance criteria and Corrective Action: Surrogate recoveries are calculated on all samples, blanks and spikes. The recoveries for o-Terphenyl are compared to the method acceptance limits. The recoveries for n-Triacontane-d_{62} are compared to nominal acceptance limits of 70-130% until laboratory acceptance limits can be established.

When a sample has a surrogate that falls outside of the method acceptance limit window, the problem should be investigated. If the recovery looks like it is affected by the sample matrix, the sample may be reinjected to confirm matrix interference. When a sample has no detectable surrogate recovery, the sample should be reextracted.

8.6 CAR: Whenever data is not acceptable because of a failing LCS or surrogate recovery, a corrective action report (CAR) must be initiated as soon as possible.

9.0 METHOD PERFORMANCE

9.1 The MDL of this method is estimated to be at least 4 mg/kg for soil and 0.1 mg/L for water. Each laboratory shall establish a laboratory specific MDL for all matrices prior to analyzing any samples.

9.2 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Organic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit and Instrument Detection Limit Studies, for procedures on determining the MDL.

Refer to the current revision of the Florida Department of Enviromnental Protection Method for Determination of Petroleum Range Organics (Method # FL-PRO) for other method performance parameters and requirements.

TITLE: **DETERMINATION OF PETROLEUM RANGE ORGANICS (PRO) BY FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION METHOD # FL-PRO**

10.0 APPLICABLE DOCUMENTS/REFERENCES

Florida Department of Environmental Protection, Method for Determination of Petroleum Range Organics, Method # FL-PRO, Revision 1, November, 1995.

ASTM "Standards Methods for Comparison of Waterborne Petroleum Oils by Gas Chromatography," 3328-78.

Wisconsin DNR Modified DRO method, July 1993, Revision 6.

"Test Methods for the Evaluation of Solid Waste: Physical/Chemical Methods", SW-846, third Edition, Final Update III, December 1996, Methods 8000B, 8100, 3500B, 3510C, 3520C, 3540 and 3550B.

LIST OF TABLES AND FIGURES

TITLE: **DETERMINATION OF PETROLEUM RANGE ORGANICS (PRO) BY FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION METHOD # FL-PRO**

TABLE 1

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Method blank	One per prep batch	No analyte detected >PQL	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e. If the blank results are above the PQL, report sample results which are < PQL or > 10X the blank concentration. Otherwise, reprep a blank and the remaining samples.
LCS	One per prep batch	Laboratory established acceptance limits	(1) Evaluate the samples and associated QC: i.e. If an MS/MSD was performed and acceptable, narrate. If an LCS/LCSD was performed and only one of the set was unacceptable, narrate. If the surrogate recoveries in the LCS are low but are acceptable in the blank and samples, narrate. If the LCS recovery is high but the sample results are < PQL, narrate. Otherwise, reprep a blank and the remaining samples.
Initial Calibration	Initial cal prior to sample analysis	Correlation coefficient => 0.995	(1) Perform instrument maintenance as needed. (2) Reanalyze and or reprep calibration standards.
CV(At or near the midpoint of the ICAL)	On each working day prior to sample analysis if an ICAL was previously analyzed	± 25 %D	(1) Evaluate the samples: If the %D>+25% and sample results are <PQL, narrate. If %D>±25% and is likely a result of matrix interference, narrate. All samples must be reanalyzed that fall within the standard that exceeded criteria and the last standard that was acceptable.
End of sequence CV	At the end of each 12-hour work shift or after running 10 samples, whichever is sooner	± 25 %D	(1) Evaluate sample data if criteria exceeded due to matrix; narrate, and perform maintenance for new samples. (2) If criteria are exceeded and this is not due to matrix, Reanalyze.
Matrix Spike/Matrix Spike Duplicate	One for every set of 20 samples provided samples aliquots are not depleted	Laboratory established acceptance limits RPD< 20 % for waters and < 25 % for solids	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS/MSD are unacceptable, reprep the samples and QC.
Sample Duplicate (If required in lieu of MSD)	One sample duplicate per twenty samples	RPD ≤20 for waters, RPD ≤25 for solids	(1) Evaluate data for matrix interference homogeneity of sample.
Demonstration of analyst proficiency; accuracy and precision	One time per analyst initially and annually thereafter	Must pass all applicable QC for method	Repeat analysis until able to perform passing QC; document successful performance in personal training file

TITLE: **DETERMINATION OF PETROLEUM RANGE ORGANICS (PRO) BY FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION METHOD # FL-PRO**

TABLE 1 (cont.)

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Demonstration of analyst proficiency; accuracy and precision	One time per analyst initially and annually thereafter	Must pass all applicable QC for method	Repeat analysis until able to perform passing QC; document successful performance in personal training file
MDL study	Once per year	Ideally, PQL = at least 3 x MDL	Repeat MDL

TITLE: **DETERMINATION OF PETROLEUM RANGE ORGANICS (PRO) BY FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION METHOD # FL-PRO**

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-333-04	FL-PRO, current revision
Apparatus/Materials		
Reagents	5.3 Sodium sulfate purified by vendor	7.3 Sodium sulfate purified by heating at 400 deg C for 4 hours or extracting x3 with methylene chloride and drying at 105 deg C
Reagents	5.5 PRO free sand (Muffled)	3.4 Ottawa sand
Sample preservation/ handling		
QC – Method Blank	Table 1 No analyte detected >PQL	10.4 ...TRPH value of the blank shall be at or below the established method detection limit.
QC - Surrogates	Use n-Triacontane-d_{62}. Use nominal limits of 70-130 until laboratory limits can be established.	7.4.1 Recommend C_{39}. Use method acceptance limits.
QC - Spikes	8.2 ...PRO concentration of 850 ug/L for waters and 28.5 mg/kg for soils.	7.4.4 Total PHS concentration in the spiked sample of 5 mg/L in water or 300 mg/kg in soils. ...The concentration of the spike in the sample should be approximately 3-5 times the level expected in the sample...level of the spike should be adjusted...
QC - Accuracy/Precision		
QC – LCS		
QC - MDL		
Procedure	7.4.2.2 Retention Time Window – Establish daily retention time windows for each analyte of interest using the absolute retention time for each analyte as the midpoint of the window for that day **if** after analyzing the midpoint it is determined that one or more analytes falls outside of the previously established absolute retention time window. The daily retention time window equals the midpoint ± three times the standard deviation determined in Section 7.3.	9.3.2.2 Retention Time Window – The retention time window for the surrogates and C8 and C40 shall be within the established range ... If they are out of acceptance range, a new initial calibration must be prepared and verified before samples are analyzed.

TITLE: **DETERMINATION OF PETROLEUM RANGE ORGANICS (PRO) BY FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION METHOD # FL-PRO**

TABLE 2, cont'd

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-333-04	FL-PRO, current revision
Procedure	7.5.4 The Methylene chloride blank is analyzed if requested by the client and will be less than the PQL.	9.5.4 The methylene chloride blank must be analyzed with each sequence and the PRO concentration shall be less than the MDL of the method.
Procedure	7.5.4 Baseline correction is allowed to correct for rises due to temperature programming. Range integration is corrected by the automatic subtraction of the baseline established by activation of a programmed run without the injection of any material. Instrument baseline must be established for every batch of samples.	9.5.4 Do not baseline subtract
Procedures	5.7 The standards are prepared at the following five different concentrations: 200 ug/ml, 100 ug/ml, 50 ug/ml, 20 ug/ml, and 5 ug/ml (per each component).	7.4.3 Suggested calibration levels are 5, 50, 150, 250, 350 and 500 ug/mL of each individual component.

TITLE: **DETERMINATION OF PETROLEUM RANGE ORGANICS (PRO) BY FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION METHOD # FL-PRO**

TABLE 3

METHOD ACCEPTANCE CRITERIA

	% Recovery			Precision (%RSD)		
	Water	Soil		Water	Soil	
		Soxhlet	Sonication		Soxhlet	Sonication
Matrix Spike Samples	41-101	41-224	62-204	0-20	0-25	0-25
Laboratory Control Spike Samples	55-118	63-135	63-153	0-20	0-25	0-25
Surrogates: OTP	82-142	57-115	62-109			
n-triacontane-D_{62}	70-130	70-130	70-130			

TABLE 4

PQLS

Analyte	Water ug/L	Soil mg/Kg
PRO	500	20

TITLE: **DETERMINATION OF PETROLEUM RANGE ORGANICS (PRO) BY FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION METHOD # FL-PRO**

FIGURE 1

EXAMPLE OF RUNLOG PAGE

Katahdin Analytical Services, Inc.
Instrument: GC12 (FID)
Amount Injected 1ul

GC Laboratory Instrument Runlog

Method (circle): EPH(MADEP) / FL PRO / TNRCC 1005

Reviewed by/ Date:

DRO/TPH - 8015Mod. / MDEP 4.1.25 / 8100Mod.

Date	Init.	Result File	Sample ID	Y/N	Method	Column	Comments
030508	KGS	CK2029	SD 5ug/ml	Y	AKROJ15A	289	
		30	10 5ug/ml	Y			
		31	1 5ug/ml	Y			
		32	AKO END SD	Y			
		33	WG45750-1 BLK	Y			cont
		34	SB05Dz-4	Y			
030608		35	Mech	A	ALIBOZZA		
		36	Thermal Blank	N			
		37	PHC SD 1ug/ml	Y			Concentrated per Emp.
		38	PRO SD 1ug/ml	Y	AKROJ15A		
030708	KGS	39	Thermal Blank	N	FLPO1EA	289	Aliner
		40		N			
		41		Y			
		42	FLPSD 1ug/ml	Y			H1709
		43	200	Y			H1710
		44	100	Y			H1712
		45	20	Y			H1713
		46	5	Y			H1711
		47	END SD 1ug/ml	Y			H1714
		48	WG48607-1 SD	Y			
		49	-2	Y			
		50	-3	Y			
		51	SB0564-1	Y			
		52	-2	Y			
		53	Mech	Y			
		54	Thermal Blank	Y			
		55	FLPSD 1ug/ml	Y			H1709
031008	KGS	59	TB	N	ALJ6Q12A		
		60		Y			
		61	PHC SD 1ug/ml	Y			

QAGC186

0000052

TITLE: **DETERMINATION OF PETROLEUM RANGE ORGANICS (PRO) BY FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION METHOD # FL-PRO**

FIGURE 2

EXAMPLE OF DATA REVIEW CHECKLIST

Verbal Due Date _____ Due Date_____

Client:		Primary	Secondary
Method:		Date:	Date:
SDG No:	Level:	Initials:	Initials:
KAS No:			Approved : ☐ Yes

PRIMARY REVIEW CHECKLIST

Highlight Method / project specific information. _____

All needed forms are present . _____

Sample Data Summary Included (Level III & IV). _____

Correct Work Order Number or SDG name (all forms). _____

Correct project name and spelling (all forms). _____

Correct file numbers (all forms). _____

Analysis Date Correct. _____

Extraction Method & Analysis Method Correct. _____

Product list compared to ROAs (compounds & PQLs). _____

Chromatogram reviewed for unlabeled peaks (check product list). _____

Flagging of all ROAs correct (Florida Flagging ☐). _____

All tunes included (level IV) . _____

All log book pages included (Soil weights,TCLP & SPLP). _____

Verify quant results for CLP. _____

Update sample history files. _____

Sign & Date Manual integration (Narrate as needed). _____

Sample I.D's Truncated (NARRATE). YES ☐ Please list KAS # below :

_____

First correction ☐ → Review and replace appropriate SDS Forms ☐.

Second correction ☐ → Review and replace appropriate SDS Forms ☐.

Last saved by kasgc04/13/07

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-500
Revision History
Cover Page
Page 1

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS

Prepared By: Mike Thomas Date: 7/96

Approved By:

Group Supervisor: Michael F. Thomas Date: 11/15/00

Operations Manager: [signature] Date: 10/25/00

QA Officer: Deborah J. Nadeau Date: 10.23.00

General Manager: Duncan F. Hughes Date: 11/16/00

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Minor changes throughout. Clarifications to procedure section.	DN	10.23.00	10/23/00
02	removed references to medium level extraction. New logbook figures Minor changes throughout	LAD	020305 ~~02~~ LAD 020305	020305
03	updated compound list changes in wording to clarify updated logbook	LAD	04/06	04/06
04	Added definitions, added waste information, added LCSD, updated solvent exchange, updated Table 1, replaced fig. 2, added PCB cleanup to Sect. 2	LAD	09/07	09/07
05	Updated LB example. Added temp. of nitrogen water bath, lot numbers of filter paper, lot #'s of acids need to be recorded in LB. Change "N-Lo" waste to "K" waste.	LAD	07/08	07/08

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Added requirement to add spike before Na_2SO_4. Changed N_2 waterbath temperature from < 37°C to < 30°C. Removed respirator references. Added KAEHS manual, Added CA-108 reference for subsampling	LAP	02/09	02/09

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-500-06**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-500-06**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS.**

Recipient: _____ Date: _____

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures and requirements for the preparation of solid samples for pesticides/PCBs analysis in accordance with SW-846 Method 3550, current revision..

1.1 Definitions

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of, analysts experienced in the extraction of sediment/soil samples for pesticides/PCBs analysis. Each analyst must demonstrate the ability to generate acceptable results with this method.

It is the responsibility of all Katahdin personnel involved in the preparation of solid samples for pesticides/PCBs analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also

be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for the data.

It is the responsibility of the Department Manager to oversee that the members of his/her group follow this SOP, that their work is properly documented, and to indicate periodic review of the associated logbooks.

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials, and appropriate segregation of hazardous wastes. Everyone involved with the procedure must be familiar with the material safety data sheets for all the materials used in this procedure. Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Waste Disposal

Wastes generated during the preparation of samples must be disposed of in accordance with the procedures described in the current revision of the Katahdin Analytical Environmental Health and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the rinsing of glassware etc. should be disposed of in the “D” waste stream satellite accumulation area nearest

the point of generation. Acetone and hexane are considered flammable waste, and should be disposed of in the "O" waste stream satellite accumulation area nearest the point of generation. Post-extraction soil samples, used glass wool, and sodium sulfate waste should be disposed of in the soil with organics "I" waste stream satellite accumulation area nearest the point of generation. Acid waste generated during the cleanup of PCB samples should be disposed of in the "K" satellite accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

2.0 SUMMARY OF METHOD

Pesticides/PCBs are extracted from solid samples by sonication with a methylene chloride/acetone solution (1:1 by volume) following EPA Method 3550, current revision. The resulting extract is dried, concentrated, and solvent exchanged to hexane for analysis by GC. Sulfuric acid cleanup is performed on extracts for 8082 PCB analysis.

This SOP applies to low level extraction of pesticide/PCB pollutants from solid sample matrices.

3.0 INTERFERENCES

Solvents, reagents, glassware, and other sample preparation apparatus may yield interferences to GC analysis due to the presence of contaminants. These contaminants can lead to discrete artifacts or elevated baselines in chromatograms. Routinely, all of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running reagent blanks. Interferences caused by phthalate esters can pose a major problem in pesticide analysis. Common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory. At no time may gloves that have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Whenever possible, plastic items in this lab, must be replaced with metal, teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure clean glassware and apparatus are used, pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis.

Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to inteferences, further cleanup of the sample extract may be needed to minimize interferences.

4.0 APPARATUS AND MATERIALS

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

Prior to use, all glassware must be rinsed three times with methylene chloride.

4.1 Beakers - 400 mL

4.2 Kuderna-Danish (KD) apparatus - Concentrator tube - 10 mL
Evaporative flask - 500 mL
Snyder column - 3-ball macro

4.3 Powder funnels, 100 mm diameter, 35 mm stem

4.4 Vacuum filtration flask - 500 mL Erlenmeyer

4.5 Buchner funnel, porcelain, Coors® with 85 mm plate diameter (or equivalent)

4.6 Sonic disruptor – Misonix XL2015 (or equivalent), equipped with dual titanium horn extenders for extracting two samples at a time.

4.7 Spatula - stainless steel

4.8 Filter paper, 18.5 cm, Fisherbrand or Whatman 114V (or equivalent)

4.9 Boiling chips - 12 mesh, silicon carbide (or equivalent)

4.10 Water bath - eight position concentric ring bath or equivalent, equipped with a calibrated thermometer

4.11 Filter paper - 7.0 cm, Whatman, #4, or equivalent

4.12 Syringe - gas tight, 1.0 mL, solvent rinsed between each use

4.13 Balance – top-loading, capable of weighing to 0.1 g

4.14 Nitrogen evaporation apparatus

5.0 REAGENTS

5.1 Sodium sulfate - (ACS reagent grade) powdered, anhydrous, certified by the manufacturer/vendor as purified by heating to 400 °C prior to receipt by the laboratory. Solvent rinse immediately before use by rinsing three times with pesticide grade methylene chloride.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

5.2 Sodium sulfate - (ACS reagent grade) granular, anhydrous, purified as described in section 5.1.

5.3 Methylene chloride - (pesticide grade or equivalent) purchased by lot, evaluated prior to use by concentration of 300mLs to 1.0 mL followed by GC/MS analysis.

5.4 Acetone and hexane - pesticide grade or equivalent

5.5 Organic-free sand, purified by baking at 400 °C for four hours. Method blanks serve as checks on the baked sand.

5.6 Surrogate spiking solution - Prepare a solution of decachlorbiphenyl (DCB) and tetrachloro-meta-xylene (TCMX) at a concentration of 1 ug/mL each in acetone. Store the solution at –10 to -20 °C (±2 °C) in a Teflon sealed container. Solution must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.

5.7 Pesticide Matrix spike/Lab control sample spiking solution - Prepare a spiking solution in pesticide grade methanol that contains all target analytes listed below:

Analyte	ug/mL
4,4'-DDD	0.5
4,4'-DDE	0.5
4,4'-DDT	0.5
Aldrin	0.5
alpha-BHC	0.5
beta-BHC	0.5
delta-BHC	0.5
Dieldrin	0.5
Endosulfan I	0.5
Endosulfan II	0.5
Endosulfan Sulfate	0.5
Endrin	0.5
Endrin Aldehyde	0.5
Endrin Ketone	0.5
gamma-BHC (Lindane)	0.5
Heptachlor	0.5
Heptachlor Epoxide	0.5
Methoxychlor	0.5
alpha-Chlordane	0.5
gamma-Chlordane	0.5

Store the solution at –10 to -20 °C (±2 °C) in a Teflon sealed container. Solution must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.

5.8 PCB Matrix Spike/Lab Control Sample Spiking Solution – Prepare spiking solution in pesticide grade acetone that contains PCB's Arochlor 1016 and Arochlor 1260 (1660), both at 5.0 ug/mL.

Store the solution at –10 to -20 °C (±2 °C) in a Teflon sealed container. Solution must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Soil samples are collected in glass soil jars and stored at 4°C (±2°C) until time of extraction.

Holding time for extraction of sediment/soil samples for Method 3550 is 14 days from date of sample collection, although the analyst should be aware that actual holding times employed may be project/program specific.

Store all extracts at 4°C (±2°C) in the dark in labeled Teflon-sealed containers. See SOP SD-902, "Sample Receipt and Internal Control," current revision, for storage areas and temperature maintenance procedures.

7.0 PROCEDURES

LOW LEVEL EXTRACTION OF SOIL/SEDIMENT FOR PESTICIDES/PCBs

The low level extraction procedure is designed for the preparation of soil/sediment samples that may contain analytes at levels lower than 20,000 ug/kg. The procedure involves extraction of pesticides and PCBs from an initial sample weight of 30.0 ± 0.1 g using an ultrasonic cell disruptor.

Many solid samples may need to be cleaned up to reduce matrix interferences. The cleanup procedure employed will be dependent upon the nature of the interferences and the target compounds to be analyzed, and options may include acid wash, sulfur cleanup, florisil cleanup, or gel permeation chromatography (GPC). The Department Manager should be consulted to determine if a particular sample should be subjected to further cleanup procedures; the decision should consider sample history, sample appearance, and project/client needs. All extracts or extract splits for subsequent 8082 PCB analysis will, at a minimum, undergo acid cleanup. (Refer to SOP CA-525, current revision)

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

7.1 Discard any excess water on the sediment sample. Mix with a stainless steel spatula to ensure homogeneity of the sample. If the sample container is full to the extent that stirring the sample is impractical, try to remove the "best representative" aliquot from the jar based on color, particle size, moisture, etc. Remove any foreign objects such as sticks, leaves, or rocks, and note actions taken in the appropriate extraction logbook. **Please refer to the current revision of Katahdin Analytical Services SOP CA-108, "Basic Laboratory Technique", for more detailed guidance on subsampling to ensure reproducibility.**

7.2 Weigh out a 30.0 ± 0.1 g portion of sample into a labeled 400 mL beaker. Record sample weight to nearest 0.1 g in appropriate extraction logbook. Refer to sections 7.6 and 7.7 for spike and surrogate addition instructions. Add between 30 g and 60 g of powdered sodium sulfate, as required, to produce a "free-flowing" mixture. The amount of sodium sulfate added will depend upon the moisture content of the sample (e.g., low moisture content will require less sodium sulfate). Mix well with a spatula. Keep the spatula in the sample beaker and cover the beaker with aluminum foil.

7.3 A method blank must be prepared with each extraction batch, not to exceed 20 client samples. To prepare method blank, weigh out one 30.0 ± 0.1 g portion of purified sand in a labeled 400 mL beaker. Refer to sections 7.6 and 7.7 for spike and surrogate addition instructions. Add 60 g sodium sulfate and mix well. (Although a "free-flowing" mixture can be achieved with less than 60 g sodium sulfate, the method blank must contain 60 g in order to evaluate the sodium sulfate as a potential source of contamination.)

7.4 A laboratory control sample (LCS) must be prepared with each extraction batch, not to exceed 20 client samples. To prepare LCS, weigh out one 30.0 ± 0.1 g portion of purified sand in a labeled 400 mL beaker. Refer to sections 7.6 and 7.7 for spike and surrogate addition instructions. Add 30 g sodium sulfate and mix well. With extraction batches prepared for combined 8081/8082 Pesticide and PCB analysis, separate Pesticide and PCB LCS's must be prepared (refer to sections 5.7 and 5.8). If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or Department Manager.

7.5 A matrix spike/matrix spike duplicate (MS/MSD) set must be prepared for every 20 samples. To prepare MS/MSD, weigh out 30.0 ± 0.1 g portions of the sample designated for MS/MSD into each of two labeled 400 mL beakers. Record sample weights to nearest 0.1 g in appropriate extraction logbook. Refer to sections 7.6 and 7.7 for spike and surrogate addition instructions. Add 30 - 60 g sodium sulfate to each to produce a free-flowing mixture, and mix well. With extraction batches prepared for combined 8081/8082 Pesticide and PCB analysis, separate Pesticide and PCB MS/MSD pairs must be prepared (refer to sections 5.7 and 5.8).

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

7.6 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL pesticide/PCBs surrogate spiking solution using a 1.0 mL gas tight syringe. The surrogate spike should be added **prior** to the addition of the sodium sulfate. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent prior to each use.

7.7 To LCS/LCSD and MS/MSD pairs add 1.0 mL of pesticide or PCB matrix spike/LCS spiking solution using a 1.0 mL gas tight syringe. The LCS/MS spike should be added **prior** to the addition of the sodium sulfate. Record matrix spike/LCS spiking solution volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent prior to spiking a different solution and when spiking is completed.

7.8 To assure optimum operation and maximum energy output, the sonicators must be tuned daily prior to extracting samples. The following tuning procedure must be performed with the sonicator probes vibrating in air.

7.8.1 Turn OUTPUT CONTROL knob counter-clockwise to zero, and turn Pulsar Duty Cycle to off (or continuous mode).

7.8.2 Press POWER SWITCH to ON (up) position. Engage the Timer Switch (W-375)

7.8.3 Press and hold down the TUNE switch.

7.8.4 Turn the Output Control Knob towards setting 10. Note the position of the needle on the % output power meter. **DO NOT exceed 70%. If you reach 70% - STOP!!** Rotate the Tuning Knob clockwise or counter-clockwise until a minimum (not maximum) reading (usually less than 20%) is obtained.

7.8.5 Turn the Output Control Knob towards setting 10. Again, note the position of the needle and do not exceed 70%. Rotate the Tuning Knob until you obtain a minimum reading of 20% or below.

7.8.6 Release the TUNE switch. **CAUTION: Do NOT touch probe. The probe is still active.**

7.8.7 Turn OUTPUT CONTROL KNOB counter-clockwise to zero (or disengage timer).

7.8.8 Confirm that the sonicators were tuned by recording the date and/or percent in the extractions logbook.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

Note: If the unit will not be used immediately, please turn the power switch to off.

7.9 Prior to extracting any samples, ensure that the sonicator probes are decontaminated by rinsing with a methylene chloride wash bottle. Collect the waste in a waste beaker. It may sometimes be necessary to wipe the upper part of each probe with a methylene chloride dampened KimWipe. Repeat this decontamination step between each sample on each probe.

7.10 To the mixed and spiked blank and LCS, add 100 mL of the 1:1 methylene chloride/acetone solution and proceed with steps 7.11 through 7.14.

7.11 It may be necessary at this time to stir the sample/sodium sulfate mixture with the spatula to loosen up the mixture prior to extracting. Position beaker in the ultrasonic cell disruptor so that the bottom surface of the tip of the 3/4 inch disruptor horn is about halfway below the surface of the solvent and above the sediment layer.

7.12 Sonicate for 3 minutes with the output control knob set at 10, and mode switch on "pulsed" and % duty cycle knob set at 50%. While the mixture is sonicating, one should be able to see all, or most of the material, moving in the beaker under the influence of the energized probes. If not, stir the mixture again.

7.13 Prepare a filter flask fitted with a Buchner funnel. The Buchner funnel should contain a 7.0 cm Whatman #4 filter; prerinse flask, funnel and filter with methylene chloride and discard rinsings into solvent waste container. Decant extract into the filter flask through Buchner funnel. A vacuum pump may be used to facilitate filtration or the extract may be gravity filtered.

Note: The lot number of the filter paper must be recorded in the extraction logbook.

7.14 Repeat the extraction two additional times using 100 mL portions of 1:1 methylene chloride:acetone. Before each extraction, make certain that the sodium sulfate is free-flowing and not a consolidated mass. As required, break up large lumps with the clean spatula. Decant the extraction solvent into the Buchner funnel after each sonication. On the final sonication, pour the entire sample contents into the Buchner funnel and rinse thoroughly with 1:1 methylene chloride:acetone to complete the quantitative transfer of the extract.

CONCENTRATION OF LOW LEVEL EXTRACTS

7.15 Rinse the K-D glassware (flask, concentration tube, and Snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride before assembling. Add two boiling chips to the K-D. Insert 18.5 cm filter papers into short stem powder funnels and add ~ 2 inches of sodium sulfate

crystals. Rinse the sodium sulfate in the assembled funnels with ~20-30 mLs of methylene chloride. Place the assembled K-D's under the funnels.

Note: The lot number of the filter paper must be recorded in the extraction logbook.

7.16 For a solvent exchange, add approximately 50 mL Hexane to funnel and let drain through. Since methylene chloride has a lower boiling point than Hexane, this will result in a final extract in hexane only.

7.17 Transfer the extracts to the K-D concentrator setups through the sodium sulfate in the funnels. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to pouring through the sodium sulfate. After pouring all of the extract volume through the sodium sulfate, rinse the extract flask three times with ~ 2 – 3 mLs of methylene chloride. Add the rinsings through the sodium sulfate to complete a quantitative transfer. Rinse the sodium sulfate with ~ 15 mLs of methylene chloride and allow to drain.

7.18 If samples are to be GPC'd, refer to the current revision of Katahdin SOP CA-513, Extract Cleanup Using Gel Permeation Chromatography, for appropriate concentrating procedures.

7.19 If samples are not to be GPC'd follow Steps 7.19 through 7.23 to concentrate extracts to final volume of 10.0 mLs. Otherwise proceed to GPC cleanup procedure as described in the current revision of Katahdin SOP CA-513.

7.20 Transfer the labels from the extract flasks to the K-Ds. Remove the funnel and attach a 3-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride.

7.21 Place the K-D in a hot water bath (75-85°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 6 mL, remove the K-D from the water bath. Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of methylene chloride. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL methylene chloride.

7.22 Reduce the extract in the concentrator tube to approximately 10 mL using the nitrogen blow-down apparatus. The bath temperature must be no higher than 30° C. Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-

evap sparging pipet must be rinsed down at least once or twice with ≈1 mL of methylene chloride. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N_2 sparging pipet closer to the surface of the extract to expedite the concentration. Note any problems or extract losses, if they occur, in the extractions logbook.

Note: The temperature of the water in the nitrogen evaporation water bath must be recorded in the logbook.

7.23 Complete quantitative transfer of the extract to a vial by using hexane. Adjust the volume of the hexane extract to 10 mL in either a 12 or 16 mL vial using the appropriate "reference vial" for volume comparison.

7.24 Transfer the sample label from the concentrator tubes to the vials. Store extract vials at a temperature of 4 ± 2 °C until ready for analysis. Indicate in the extractions logbook the box number and "tray location" of the individual extract vials.

7.25 All sample extracts for 8082 PCB analysis must undergo a sulfuric acid wash (cleanup) prior to analysis. All sample extracts for 8081 pesticide analysis do not undergo further cleanup unless requested by the client. All sample extracts for combined 8081/8082 analyses must be split. One portion must be acid cleaned for 8082 analysis. The associated method blank must be split and acid-cleaned in the same fashion. Prior to splitting, contents of vial must be shaken well. PCB LCSs and matrix spikes are acid cleaned also. Pesticide LCSs and matrix spikes are not subjected to further cleanup. Please refer to Katahdin SOP CA525 (current revision), Extract Cleanup Using Sulfuric Acid, for further instructions.

Note: The lot number of the acid used in PCB cleanup must be recorded in the extraction logbook.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Each extractions analyst must demonstrate proficiency in performing the extractions that prepare samples for analysis. Demonstration consists of preparation (extraction), by the analyst, of at least four aliquots which are then analyzed according to the analytical method in question. These QC samples must meet all quality control acceptance limits. Demonstration must be documented by use of a form which summarizes the results of the analysis of these aliquots, calculated percent recoveries, and standard deviation. Demonstration of proficiency must be done one time per analyst initially and then annually thereafter. Refer to SOPs QA-805 and QA-807, current revision.

If, upon analysis of the extracted samples, it is discovered that quality control acceptance criteria have not been met, all associated samples must be evaluated against all the QC. In some cases data may be reported, perhaps with narration, while in other cases, other corrective action may be taken. The corrective actions may include re-extraction of the samples associated with the quality control sample that did not meet acceptance criteria, or may include making new reagents and standards if the standardization is suspect. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 A method blank must be extracted for each and every item listed below:

- Each extraction method
- Every extraction batch of twenty or fewer samples
- Each analysis (pesticides and/or PCBs)

8.2 A laboratory control sample (LCS) is required for each and every item listed below:

- Each extraction method
- Every extraction batch of twenty or fewer samples
- Each analysis (pesticides and/or PCBs)

Refer to the current revision of the applicable Katahdin SOP for analysis of Pesticides and PCBs for quality control acceptance criteria.

9.0 METHOD PERFORMANCE

Refer to the applicable analytical SOP.

10.0 APPLICABLE DOCUMENTS/REFERENCES

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

Test Methods for Evaluating Solid Waste - Physical/Chemical Methods, Method 3550C, USEPA SW-846, Third Edition, Final Update IV, February 2007.

Table 1 Summary of Method Modifications
Figure 1 Example of Pest./PCB Soil Sample Prep Logbook Page

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

TABLE 1

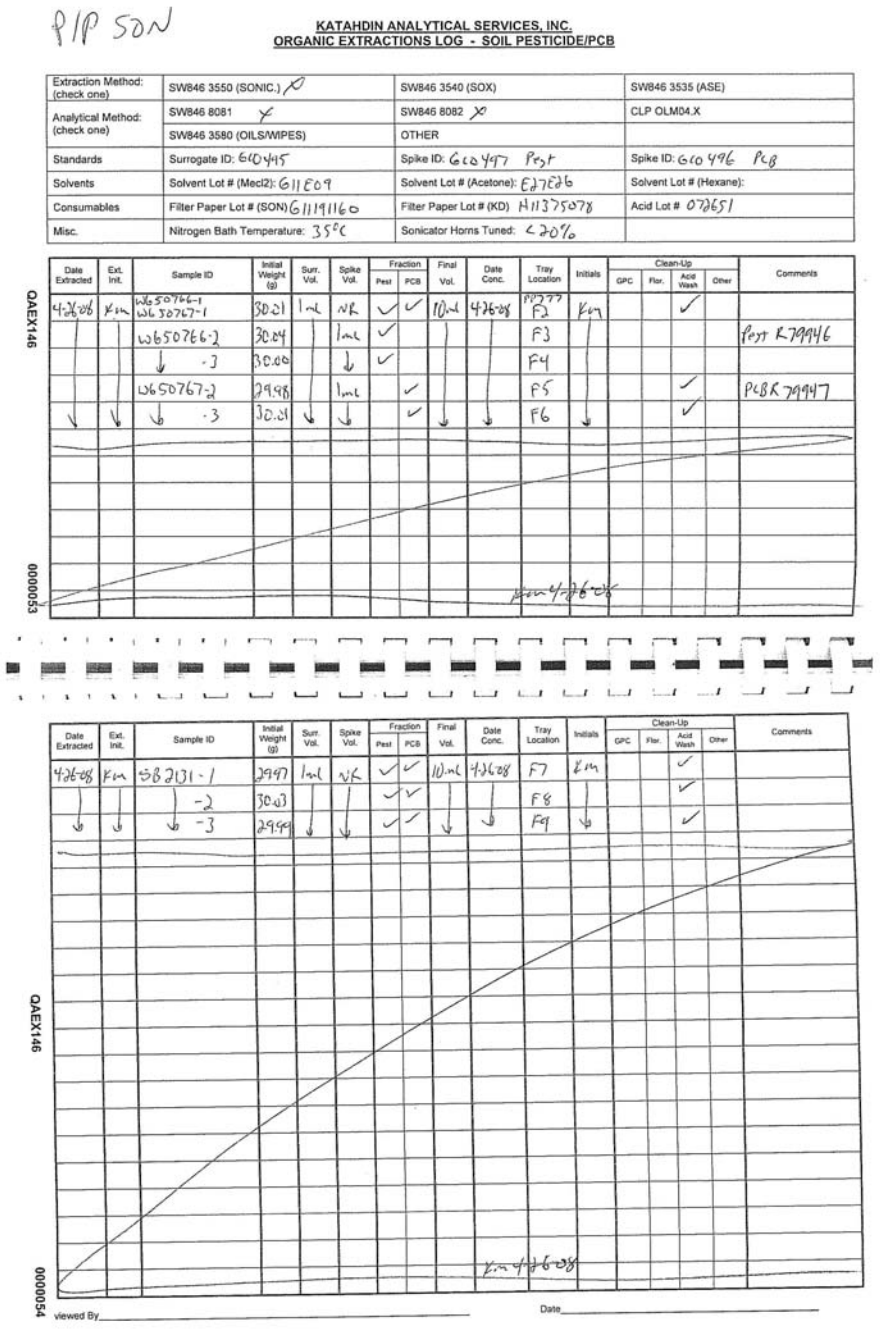
SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-500-06	METHOD 3550, current revision
Apparatus/Materials	1. short stem funnels	1. drying columns
Reagents		
Sample preservation/ handling		
Procedures	1. extract dried using Na_2SO_4 in short stem funnels 2. place sonicator horns ½ way between the surface of the solvent and the sediment layer 3. no apparatus height specification for concentration on water bath 4. water bath at 75-85 deg C 5. sample removed from water bath when volume reaches ~6 mL 6. Solvent exchange to hexane is performed using K-D apparatus with addition of approximately 50 mL hexane at the start of concentration process	1. extract dried using Na_2SO_4 in drying columns 2. place sonicator horns ½ inch below the solvent surface but above sediment layer 3. partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-15min 4. water bath at 80-90 deg C 5. sample removed from water bath when volume reaches 1-2 mL 6. Solvent exchange to hexane is performed using K-D apparatus with addition of approximately 50 mL hexane after concentrating methylene chloride extract to 1 mL
QC - Spikes	Refer to analytical SOP	
QC - LCS		
QC - Accuracy/Precision		

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

FIGURE 1

EXAMPLE OF LOGBOOK PAGE



P/P SON

KATAHDIN ANALYTICAL SERVICES, INC.
ORGANIC EXTRACTIONS LOG - SOIL PESTICIDE/PCB

Extraction Method: (check one)	SW846 3550 (SONIC.) ✓	SW846 3540 (SOX)	SW846 3535 (ASE)
Analytical Method: (check one)	SW846 8081 ✓	SW846 8082 ✓	CLP OLM04.X
	SW846 3580 (OILS/WIPES)	OTHER	
Standards	Surrogate ID: 6C0495	Spike ID: 6C0497 Pest	Spike ID: 6C0496 PCB
Solvents	Solvent Lot # (MeCl2): 611E09	Solvent Lot # (Acetone): E27E86	Solvent Lot # (Hexane):
Consumables	Filter Paper Lot # (SON) 611191160	Filter Paper Lot # (KD) H113750781	Acid Lot # 072651
Misc.	Nitrogen Bath Temperature: 35°C	Sonicator Horns Tuned: < 20%	

Date Extracted	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol.	Spike Vol.	Fraction Pest	Fraction PCB	Final Vol.	Date Conc.	Tray Location	Initials	Clean-Up GPC	Clean-Up Flor.	Clean-Up Acid Wash	Clean-Up Other	Comments
4-26-08	KM	W650766-1	30.01	1mL	NR	✓	✓	10mL	4-26-08	P1777 F2	KM			✓		
		W650766-2	30.04	1mL		✓				F3						Pest K79946
		↓ -3	30.00	↓		✓				F4						
		W650767-2	29.98	1mL			✓			F5				✓		PCBK79947
		↓ -3	30.01	↓			✓			F6				✓		

QAEX146 0000053

Date Extracted	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol.	Spike Vol.	Fraction Pest	Fraction PCB	Final Vol.	Date Conc.	Tray Location	Initials	Clean-Up GPC	Clean-Up Flor.	Clean-Up Acid Wash	Clean-Up Other	Comments
4-26-08	KM	SB2131-1	29.97	1mL	NR	✓	✓	10mL	4-26-08	F7	KM			✓		
		↓ -2	30.03			✓	✓			F8				✓		
		↓ -3	29.99	↓	↓	✓	✓	↓	↓	F9	↓			✓		

QAEX146 0000054

Viewed By _____ Date _____

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

Prepared By: Michael Thomas Date: 07-24-00

Approved By:

Department Manager: [signature] Date: 6-23-06

Operations Manager: Deborah J. Nadeau Date: 6.23.06

QA Officer: Susie Dimond Date: 6-23-06

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
03	Changes to sect. 5.5 & Figures 3 & 4 to reflect current spike solutions and concentrations Replaced cover page. Original cover page filed with SOP CA502-02	LAD	04/06	04/06
04	Added definitions, added waste information added LCSD, added SIM LCS/D, MS/D, updated Table 1, added use of narrow range pH paper. Minor changes throughout to reflect current practice.	LAD	09/07	09/07
05	Removed MS/MSD 14 day requirement. Changed CLLE extraction time to 18 → 24 hours. Added information on determining initial sample volume. Added extracted sample disposal. Removed all references to method 625.	LAD	09/08	09/08
06	Added to check pH after B/N CLLE extraction to ensure pH ≥ 11. If not add more NaOH and continue extracting. Added information for initial volume determination. Added Reference to CA-109. Updated logbook example. Added if extract goes dry - re extract.	LAD	10/09	10/09

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

Revision History (cont.):

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-502-06**, titled **PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-502-06**, titled **PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS**.

Recipient: _____ Date: _____

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe procedures utilized by Katahdin Analytical personnel in the preparation of all non-CLP aqueous samples for analysis of extractable semivolatile organic compounds.

The goal of this procedure is to ensure uniformity involving the preparation of samples for subsequent SVOA analysis by GC/MS. This SOP is applicable to EPA Methods 3510 (modified separatory funnel extraction) and 3520 (continuous liquid-liquid extraction), current revisions.

1.1 Definitions

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the extraction of samples for semivolatile analysis. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

It is the responsibility of all Katahdin technical personnel involved in the extraction of samples for semivolatile analysis to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their department follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDS's for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

Any methylene chloride solvent waste generated during the rinsing of glassware, disassembly of CLLEs after extraction, etc. should be disposed of in the "D" waste stream satellite accumulation area nearest the point of generation. Acetone and methanol are considered flammable waste, and should be disposed of in the "O" waste stream satellite accumulation area nearest the point of generation. Post-extraction aqueous samples are considered either N-Hi or N-Low waste and should be disposed of in the corresponding satellite waste accumulation area nearest the point of generation. Sodium sulfate used for sample drying should be disposed of in the soil with organics "I" waste stream satellite accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

2.0 SUMMARY OF METHOD

For aqueous samples extracted by CLLE, a one liter aliquot of sample is adjusted to pH $\leq$ 2 and extracted with methylene chloride using a continuous liquid-liquid extractor. The pH is then adjusted to pH $\geq$ 11 and the sample is extracted again with methylene chloride. A modified separatory funnel extraction may also be used. If this procedure is used, the sample aliquot is first adjusted to pH $\geq$ 11 and then to pH $\leq$ 2. The methylene chloride extract is dried and concentrated to a volume of 1.0 mL.

3.0 INTERFERENCES

Solvents, reagents, glassware, and other sample preparation apparatus may yield interferences to GC/MS analysis due to the presence of contaminants. These contaminants can lead to discrete artifacts or elevated baselines in the total ion current profiles (TICPs). Routinely, all of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running reagent blanks. Interferences caused by phthalate esters can pose a major problem in semivolatile analysis. Common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory. At no time may gloves that have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Additionally, whenever possible plastic items in this lab must be replaced with metal or teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure that clean glassware and apparatus are used and pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis.

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to inteferences, further cleanup of the sample extract may be needed to minimize interferences.

4.0 APPARATUS AND MATERIALS

Brand names and catalog numbers are included for illustration purposes only.

4.1 Continuous liquid-liquid extractors - including body, 500 mL round bottom flask and Alhin condensers and equipped with Teflon or glass connecting joints requiring no lubrication (Hershberg-Wolf Extractor, Ace Glass Company, Vineland, NJ, P/N 6841-10 or equivalent).

4.2 Glass powder funnels.

4.3 Fluted filter paper, 18.5cm diameter.

4.4 Concentrator tube - Kuderna-Danish, 10 mL, graduated (Kontes K-570050-1025 or equivalent). Calibration must be checked at the volumes employed in the test.

4.5 Evaporation flask - Kuderna-Danish, 500 mL (Kontes K-570001-0500 or equivalent). Attach to concentrator tube with neck clips.

4.6 Snyder column - Kuderna-Danish, three- or four-ball macro (Kontes K-503000-0121 or equivalent).

4.7 Syringe - gas tight, 1.0 mL, solvent rinsed between each use.

4.8 Vials - Glass, 1.8 mL capacity, with polytetrafluoroethylene (PTFE)-lined screw top and 12 mL with Teflon-lined caps.

4.9 2 L separatory funnel, equipped with Teflon stopper and stopcock; Nalgene Teflon FEP separatory funnels may also be used.

4.10 Organic Free Boiling Chips - approximately 10/40 mesh, Teflon or silicon carbide (or equivalent). Cleaned by Soxhlet for 18 hours.

4.11 Water bath - heated, with concentric ring cover, capable of temperature control (± 20°C). The bath should be used in a hood.

4.12 Nitrogen evaporation apparatus.

4.13 Wide range pH test strips, pH 0-14, Whatman CF Type.

4.14 Glass rods for stirring samples.

4.15 Amber bottles or other appropriate containers for collection of extracts from separatory funnel extraction.

4.16 5 ¾" Pasteur pipets.

4.17 Narrow range pH test strips, pH 0 to 2.5 pH, EMD ColorpHast or equivalent.

4.18 Narrow range pH test strips, pH 11 to 13 pH, EMD ColorpHast or equivalent.

5.0 REAGENTS

All reagent and solvent lots must be checked for possible contamination. Refer to the current version of Katahdin SOP CA-105, Reagent and Solvent Handling, for further details. The extraction staff is responsible for submitting samples to the GC or GC/MS sections for appropriate analysis. All information concerning preparation of the reagent/solvent lot sample will be recorded in the Organic Extraction Log (Figure 1) and acceptance or rejection of these lots must be recorded in the solvent/reagent lot check logbook (Figure 2). All reagents and solvents must be free (<PQL) of any target compounds.

5.1 Laboratory Reagent Grade Water - defined as water in which an interferent is not observed at or above the PQL of each parameter of interest. Deionized water filtered through activated charcoal.

5.2 Sodium sulfate - granular. Bake at 400°C for 4 hours (may be done by vendor). Purify by rinsing three times with pesticide grade methylene chloride. Allow residual methylene chloride to evaporate before each use. Cool in a desiccator and store in a glass bottle with a Teflon-lined cap.

5.3 Sulfuric acid solution (1:1 H_2SO_4 : H_2O) - slowly add 500 mL of H_2SO_4 (sp gr 1.84) to 500 mL reagent water.

5.4 Acetone, methanol, methylene chloride - pesticide residue analysis grade or equivalent.

5.5 Standard Preparation - For all standard preparations, see current revision of the following Katahdin Analytical SOPs:

- "Standards Preparation, Documentation and Traceability", (CA-106, current revision)
- "Balance Calibration," (CA-102, current revision)

5.5.1. Base/Neutral and Acid (SVOA) Surrogate Spiking Solution - Surrogate standards are added to all samples and calibration solutions. Prepare a surrogate standard spiking solution that contains the following compounds at the indicated concentrations in acetone.

Compound	Conc.
phenol-$_{d6}$	100 ug/mL
2,4,6-tribromophenol	100 ug/mL
2-fluorophenol	100 ug/mL
nitrobenzene-$_{d5}$	50 ug/mL
p-terphenyl-$_{d14}$	50 ug/mL
2-fluorobiphenyl	50 ug/mL

Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem. If reanalysis of a method blank still indicates surrogates out of criteria, a new surrogate solution must be used.

5.5.2 SIM Surrogate Spiking Solution- Surrogate Standards are added to all samples and calibration solutions. Prepare a surrogate solution that contains the following compounds at a concentration of 2 ug/mL in acetone.

Compound	Conc. ug/mL
Fluorene-d10	2.0 ug/mL
2-Methylnaphthalene-d10	2.0 ug/mL
Pyrene-d10.	2.0 ug/mL
2,4-Dibromophenol	2.0 ug/mL

Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem. If reanalysis of a method blank still indicates surrogates out of criteria, a new surrogate solution must be used.

5.5.3 SVOA Matrix Spike/Lab Control Samples Spiking Solution - the matrix spike/LCS solution consists of the compounds listed in Figure 3.
Prepare a spiking solution that contains each of the base/neutral compounds listed in Figure 3 at 50 ug/mL in methanol and the acid compounds at 100 ug/mL in methanol. Matrix spike/LCS standards are stored in the freezer (-10°C to -20°C) located in the storage area.

5.5.4 Base/Neutral (SIM) Matrix Spike/ Lab Control Sample Spike Solution for SIM-SVOA. Prepare a spiking solution in methanol that contains the compounds listed in Figure 3 at a concentration of 2 ug/mL for base/neutral.

Take out 1.0 mL of Base/Neutral and Acid Matrix Spike/Lab Control Spiking Solution for SVOA and dilute it to 25.0 mL of methanol. Store the solution Spiking at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

5.5.5 Base/Neutral and Acid (SVOA) Appendix IX Lab Control Sample / Matrix Spike Spiking Solution – Prepare a spiking solution in methanol that contains the compounds listed in Figure 4 at concentrations of 100 ug/ml. Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Continuous liquid-liquid (Method 3520) and/or separatory funnel (Method 3510) extractions for semivolatiles must be started within seven days of date of sample collection, although the analyst should be aware that actual holding times employed may be project/program specific. If sampling date is unknown, the hold time is counted from one day prior to date received.

7.0 PROCEDURES

The following information must be recorded in the extraction logbook.

- Extraction method
- Surrogate and spike IDs
- Lot numbers of all solvents, acids and bases, sodium sulfate, filter paper
- Nitrogen evaporation water bath temperature
- Sample pH if appicable
- Extraction and Concentration dates
- Extraction and Concentration analyst
- Sample ID or QC sample ID
- Initial and final volumes or weight
- Surrogate and spike amounts
- Any sample cleanup preformed
- Final extract tray location
- Any comments regarding the sample extraction (ie. Emulsion)
- Prep batch start time and end time
- CLLE start time and end time
- Lot number of the vials the concentrated extracts are stored in.

The internal chain-of-custody must be signed when removing and replacing samples in storage locations.

7.1 CONTINUOUS LIQUID-LIQUID EXTRACTION (Method 3520)

7.1.1 Set up the CLLE apparatus. All glassware should be pre-rinsed three times with methylene chloride in order to eliminate any contamination factors.

7.1.2 Add approximately 500 - 600 mL of methylene chloride to the CLLE body. Label each flask with the following: sample number (or QC identification number), analyte (SVOA), extraction method (CLLE), and extraction date.

7.1.3 A method blank and a laboratory control sample (LCS) must be prepared for each daily extraction batch of twenty samples or fewer (if a work order consists of more than twenty samples, a new batch must be started on a separate page with its own method blank and LCS). To prepare method blank and LCS, add 1 L reagent water to a CLLE body. Be sure that no water leaks into the round bottom flask. If combined SIM-SVOA analysis is requested, a separate LCS must be prepared for each analysis. This blank and LCS are carried through the entire extraction and analytical procedure.

7.1.4 Mark the sample level (meniscus) on the sample bottle with a wax crayon so that the volume can be measured (this may be done prior to removal from the walk-in cooler). Transfer the sample to a CLLE body, being sure that no water leaks into the round bottom flask.

7.1.5 If the batch requires a MS/MSD, transfer two 1 L portions of the sample selected/designated for MS/MSD to CLLE bodies for preparation of a matrix spike/matrix spike duplicate if required. If combined SIM-SVOA analysis is requested, a separate MS/MSD must be prepared for each analysis. If extra MS/MSD aliquots of sample are unavailable a laboratory control sample duplicate (LCSD) may be substituted.

7.1.6 Check the pH of each sample with wide range pH paper by removing a couple of sample drops with a clean disposable pipet or on the tip of a stirring rod. Adjust the pH of the samples (including method blank, LCS/LCSD, and MS/MSD) to $\leq$ pH 2 with 1:1 H_2SO_4 after addition of surrogates and spikes and prior to attaching Allihn condensers (Step 7.1.11). Stir with a glass stirring rod and check pH by tapping the glassrod onto wide range pH paper. The pH must be ≤ 2. If the pH test strip does not clearly indicate the pH is less than 2, narrow range pH paper must be used.

7.1.7 For each sample, rinse the original sample container with approximately 30 mL of methylene chloride. Add this rinse to the CLLE body.

7.1.8 Determine the initial volume of the samples by comparing the grease marking where the sample meniscus was to the reference bottle located in the lab. Record the volume and any notable characteristics (e.g. color, presence of sediment, or odor) in the extraction logbook.

7.1.9 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL base/neutral and acid (SVOA) surrogate spiking solution using a 1.0 mL or 2.5 mL gas tight syringe. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with acetone before and after each use.

7.1.9.1 If the request is for SVOA, use the SVOA Surrogate Solution (sect. 5.5.1).

7.1.9.2 If the request is for SIM, use the SIM surrogate solution (sect. 5.5.2).

7.1.9.3 If the request is for SIM-SVOA, use the SIM surrogate solution as well as SVOA surrogate solution. NOTE: Separate LCS/LCSD and/or MS/MSD are needed for each analysis and should be spiked with the appropriate surrogate.

7.1.10 To LCS/LCSD and MS/MSD add 1.0 mL base/neutral and acid (SVOA) matrix spike/LCS spiking solution using a 1.0 mL or 2.5 mL gas tight syringe. Record matrix spike/LCS spiking solution volume and identification code in extraction logbook. Thoroughly rinse syringe with methanol before and after each use.

7.1.10.1 If the request is for SVOA -
add 1.0 mL of SVOA Matrix Spike/Lab Control Samples Spiking Solution (sect 5.5.3).

7.1.10.2 If the request is for SIM -
add 1.0 mL of SVOA Matrix Spike/Lab Control Samples Spiking Solution (sect 5.5.3) and
add 1.0 mL of Base/Neutral (SIM) Matrix Spike/ Lab Control Sample Spike Solution (sect 5.5.4).

7.1.10.3 If the request is for SVOA Appendix IX, use the SVOA Appendix IX Spiking solution as well as the SVOA spiking solution -
add 1.0 mL of SVOA Matrix Spike/Lab Control Samples Spiking Solution (sect 5.5.3) and
add 1.0 mL of Base/Neutral and Acid (SVOA) Appendix IX Lab Control Sample / Matrix Spike Spiking Solution (sect 5.5.5).

7.1.11 Attach cooling water Allihn condensers, after first rinsing each 45/50 joint with methylene chloride. Turn on the heating mantles and allow the samples to extract for 18 to 24 hours. Turn off the mantles and let samples cool.

7.1.12 Detach condensers and verify that the pH is still $\leq$ 2 in the same manner mentioned in 7.1.6. If the pH has changed, more acid should be added to make the pH $\leq$ 2 and the sample extracted for several more hours.

7.1.13 Upon completion of acid extraction, allow the sample to cool. Detach condensers and add enough 10N NaOH to adjust the pH to $\geq$ 11 with stirring. Use glass stirring rods to stir and check the pH of each sample in the same manner mentioned in 7.1.6.

7.1.14 Re-attach Allihn condensers, turn on heating mantles, and allow samples to extract for 18 to 24 hours. Turn off mantles and allow samples to cool.

7.1.15 Detach condensers and verify that the pH is still $\geq$ 11 in the same manner mentioned in 7.1.6. If the pH has changed, more NaOH should be added to make the pH $\geq$ 11 and the sample extracted for several more hours.

7.1.16 Once samples are cool to the touch, the CLLE apparatus can be disassembled. The round bottom flask is removed, covered foil and placed in the interim extract refrigerator. The remaining sample in the CLLE body is poured in the "N-Hi" satellite.

Proceed to Step 7.3 for sample extract concentration procedures.

7.2 SEPARATORY FUNNEL EXTRACTION (Modified Method 3510)

If an emulsion prevents acceptable recovery or client history indicates samples may demonstrate matrix interference, then samples should be extracted by continuous liquid-liquid extraction (CLLE).

7.2.1 Rinse all glassware, including teflon separatory funnels, three times with methylene chloride prior to use.

7.2.2 Label 2 L separatory funnels and amber collection bottles clearly. Each label should include: sample number (or QC indicator number), analyte (SVOA), matrix (Aq), extraction date.

7.2.3 A method blank and a laboratory control sample (LCS) must be prepared for every 20 samples or with each extraction batch, whichever is more frequent. To prepare method blank and LCS, add 1 L reagent water to a separatory funnel. If combined SIM-SVOA analysis is requested, a separate LCS must

be prepared for each analysis. This blank and LCS are carried through the entire extraction and analytical procedure.

7.2.4 Measure the initial volume by comparing the meniscus of the sample with the reference bottle of the same bottle type. Please refer to SOP CA-108, "Basic Laboratory Technique", for the reference bottle verification procedure. Record the volume and any notable characteristics (e.g. color, presence of sediment, or odor) in the extraction logbook.

7.2.5 If the batch requires a MS/MSD, transfer two 1 L portions of the sample selected/designated for MS/MSD to separatory funnels for preparation of a matrix spike/matrix spike duplicate if required. If combined SIM-SVOA analysis is requested, a separate MS/MSD must be prepared for each analysis. If extra MS/MSD aliquots of sample are unavailable, a laboratory control sample duplicate (LCSD) may be substituted.

7.2.6 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL base/neutral and acid (SVOA) surrogate spiking solution using a 1.0 mL or 2.5 mL gas tight syringe. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with acetone before and after each use.

7.2.6.1 If the request is for SVOA, use the SVOA Surrogate Solution.

7.2.6.2 If the request is for SIM, use the SIM Surrogate Solution.

7.2.6.3 If the request is for SIM-SVOA, use the SIM surrogate solution as well as SVOA surrogate solution. NOTE: Separate LCS/LCSD and/or MS/MSD are needed for each analysis and should be spiked with the appropriate surrogate.

7.2.7 To LCS/LCSD and MS/MSD add 1.0 mL base/neutral and acid (SVOA) matrix spike/LCS spiking solution using a 1.0 mL or 2.5 mL gas tight syringe. Record matrix spike/LCS spiking solution volume and identification code in the extraction logbook. Thoroughly rinse syringe with methanol before and after each use.

7.2.7.1 If the request is for SVOA, use the SVOA Spiking Solution.

7.2.7.2 If the request is for SIM, use the SIM Spiking solution.

7.2.7.3 If the request is for SVOA Appendix IX, use the SVOA Appendix IX Spiking solution as well as the SVOA spiking solution

7.2.8 For each sample, rinse the original sample container with 60 mL of methylene chloride. Add this rinse to the separatory funnel.

7.2.9 Adjust the pH of the samples (including method blank, LCS/LCSD, and MS/MSD) to pH $\geq$ 11 with 10N NaOH after addition of surrogates and spikes. Stir with a glass stirring rod and check pH by tapping the glass stirring rod onto wide range pH paper. The pH must be $\geq$ 11. If the pH test strip does not clearly indicate the pH is greater than 11, narrow range pH paper must be used.

7.2.10 Add 60 mL of methylene chloride directly to the method blank and LCS/LCSD separatory funnels.

7.2.11 Extract the samples by shaking the funnel for two minutes, venting often, but gently, in a hood to release pressure. A mechanical shaker may be used, where samples are shaken for 3 minutes. Following each shake, allow phases to separate for at least 10 minutes. Drain the methylene chloride layer into an amber collection bottle.

7.2.12 If an emulsion forms, mechanical techniques must be employed to achieve maximum separation. Such means include swirling, centrifugation, and draining through a small separatory funnel. In certain instances, transferring the entire sample into a continuous liquid-liquid extractor (CLLE) may be the only alternative. If any such techniques are used, they must be noted in the extractions logbook, and the batch transferred to a CLLE batch with its own batch ID.

7.2.13 Add a second 60 mL aliquot of methylene chloride to the separatory funnel and extract for the second time (see 7.2.12 – 7.2.13). Collect the methylene chloride layer in the same amber collection bottle.

7.2.14 Repeat the extraction for a third time as described in 7.2.14.

7.2.15 Following the third shake, using a glass stirring rod, check the pH to ensure that it has remained at $\geq$ 11. If the pH has changed back to neutral range, it must be readjusted to $\geq$ 11 and the sample must be extracted at least one more time, adding the methylene chloride to the same amber bottle, that was previously used. If the pH has remained at a value $\geq$ 11, the pH is then adjusted to $\leq$ 2 with 1:1 H_2SO_4. Add enough 1:1 H_2SO_4 to adjust the pH to $\leq$ 2 with stirring. Use glass stirring rods to stir.

7.2.16 Add 60 mL methylene chloride and extract the samples three times in the same manner described in 7.2.11 – 7.2.13. Collect the methylene chloride layer in the same amber collection bottle used to collect the acid fraction.

7.2.17 Sample waste should be poured into the "n-lo" sattalite.

7.2.18 Proceed to Section 7.3 for extract concentration procedures.

7.3 CONCENTRATING THE EXTRACTS

For Methods 3510 and 3520, the combined fractions are concentrated to a final volume of 1.0 mL.

7.3.1 Rinse the K-D glassware (flask, concentration tube, and snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride. Add two boiling chips to the K-D prior to final rinse. Also rinse the assembled funnels, filter paper, and granular sodium sulfate used for drying the extracts.

7.3.2 Transfer the methylene chloride extract to a K-D concentrator setup through a short stem funnel filled with 1-2 inches of sodium sulfate in fluted filter paper. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to pouring through the sodium sulfate. After pouring all of the extract volume through the sodium sulfate, rinse the extract flask three times with ~ 2 – 3 mls of methylene chloride. Add the rinsings through the sodium sulfate to complete a quantitative transfer. Rinse the sodium sulfate with ~ 15 mls of methylene chloride and allow to drain

7.3.3 Transfer the label from the collection bottle or round bottom flask (for CLLE) to a K-D. Remove the funnel and attach a 3- or 4-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride.

7.3.4 Place the K-D in a hot water bath (75-85°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 6 mL, remove the K-D from the water bath. Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of methylene chloride. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL methylene chloride.

7.3.5 Reduce the methylene chloride extract in the concentrator tube to approximately 1 mL using the nitrogen blow-down apparatus. The bath temperature must be no higher than the boiling point of the solvent (39°C for

methylene chloride). Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with ≈1 mL of methylene chloride. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N_2 sparging pipet closer to the surface of the extract to expedite the concentration. Note any problems or extract losses, if they occur, in the extractions logbook.

7.3.6 Reduce each extract to slightly less than 1 mL and then, using a 5 ¾" pasteur pipet, transfer the final extract and label to a 1.8 mL vial with PTFE-lined cap.

7.3.7 If at any time during the concentration process the concentrator tube goes dry, reextraction must occur immediately.

7.3.8 Using methylene chloride for a quantitative transfer, adjust the final volume of each extract to 1 mL. Use the 1 mL oil-filled reference vial for volume comparison.

7.3.9 Store in refrigerator until GC/MS analysis.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

A method blank must be extracted for each and every item listed below:

- Each sample matrix (soil, water)
- Each day of extraction (24 hours midnight - midnight)
- Each extraction method or level
- Every 20 samples extracted in a 24-hour period

A laboratory control sample (LCS) is required for each and every item listed below:

- Each sample matrix
- Each extraction method or level
- Every extraction batch of twenty or fewer samples

Refer to the current revision of the applicable Katahdin SOP for analysis of semivolatiles for quality control acceptance criteria.

9.0 METHOD PERFORMANCE

Refer to the applicable analytical SOP.

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste-Physical/Chemical Methods, Methods 3510 and 3520 (current revisions), SW-846 Third Edition, Updates I, II, IIA, and IIB, Revised January 1995, US EPA.

LIST OF TABLES AND FIGURES

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

TABLE 1

SUMMARY OF METHOD MODIFICATIONS (METHOD 3510, current revision)

TOPIC	KATAHDIN SOP CA-502-06	METHOD 3510, current revision
Apparatus/Materials	1) 250 mL amber bottle or flask 2) 1.0 mL syringe 3) short stem funnels	1) 250 mL Erlenmeyer flask 2) 5.0 mL syringe 3) drying columns
Reagents		
Sample preservation/ handling		
Procedures	1) extract collection in amber bottle or Erlenmeyer flask 2) Add surrogate/spike to sample in CLLE 3) Extract for 3 minutes on mechanical shaker 4) extract three times at pH $\geq$ 11, then extract three times at pH $\leq$ 2. 5) extract dried using Na_2SO_4 in short stem funnels 6) Rinse the extract flask three times with ~ 2 – 3 mLs of methylene chloride then rinse the sodium sulfate with ~ 15 mLs of methylene chloride to complete a quantitative transfer 7) water bath temp 75-85 deg C 8) no apparatus height specification for concentration on water bath 9) sample removed from water bath when volume reaches ~6 mL 10) N bath temp no higher than 39 deg C	1) extract collection in Erlenmeyer flask 2) Add surrogate/spike directly to sample bottle 3) Extract by shaking vigorously for 1 - 2 minutes with periodic venting 4) extract three times at pH $\leq$ 2, then extract three times at pH $\geq$ 11. 5) extract dried using Na_2SO_4 in drying columns 6) Rinse the Erlenmeyer flask, which contained the solvent extract, with 20 - 30 mL of methylene chloride to complete the quantitative transfer 7) water bath temp 15-20 deg C above solvent boiling temp 8) partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-20 min 9) sample removed from water bath when volume reaches 1 mL 10) N bath temp 35 deg C
QC - Spikes	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - LCS	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

TABLE 1, continued

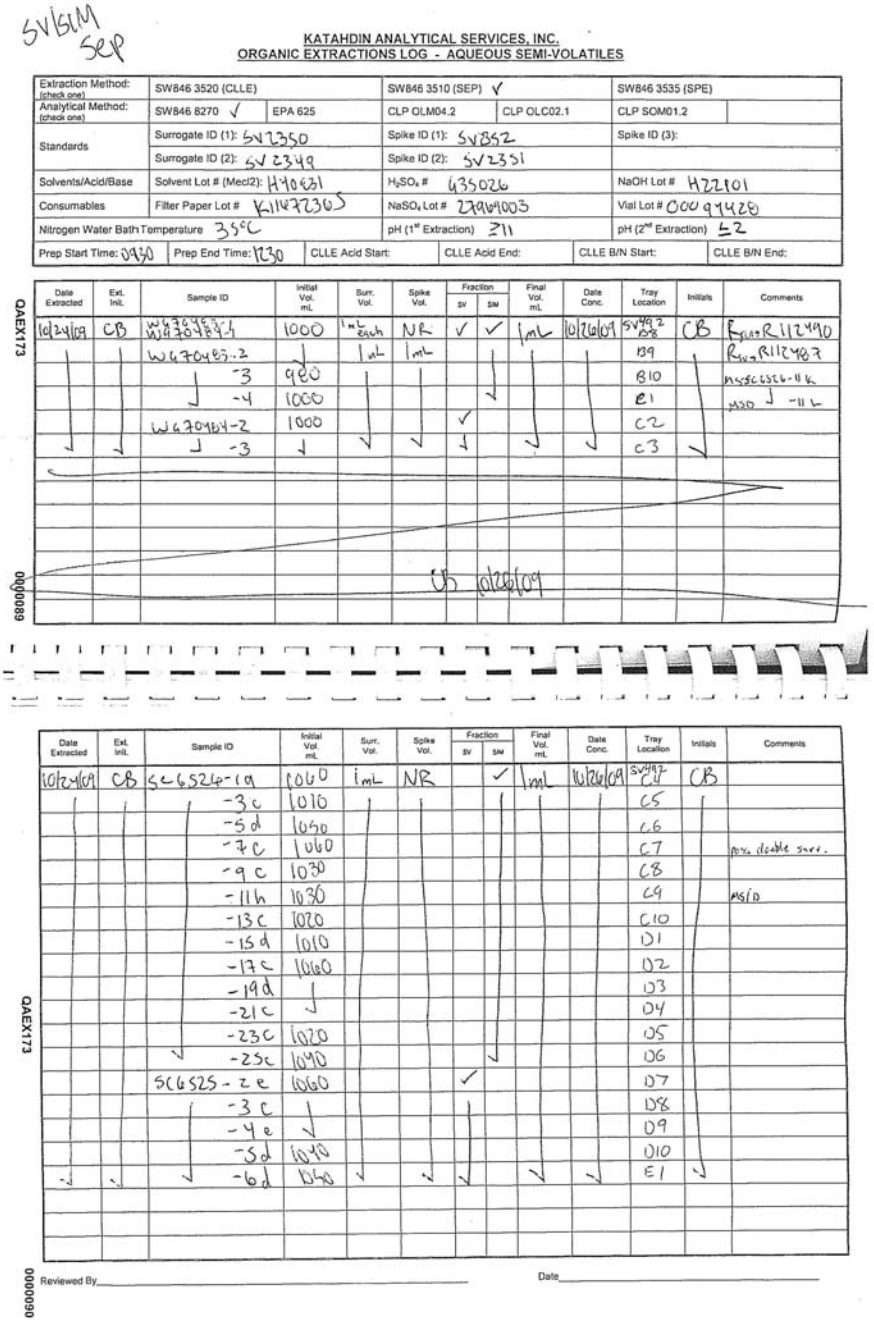
SUMMARY OF METHOD MODIFICATIONS (METHOD 3520, current revision)

TOPIC	KATAHDIN SOP CA-502-06	METHOD 3520, current revision
Apparatus/Materials	1) short stem funnels	1) drying columns
Reagents		
Sample preservation/ handling		
Procedures	1) Add surrogate/spike to sample in CLLE 2) Add approximately 500 - 600 mL of methylene chloride to the CLLE body 3) CLLE for 22 $\pm$ 2 hours 4) Extract dried using Na_2SO_4 in short stem funnels 5) Rinse the extract flask three times with ~ 2 – 3 mLs of methylene chloride then rinse the sodium sulfate with ~ 15 mLs of methylene chloride to complete a quantitative transfer 6) water bath temp 75-85 deg C 7) no apparatus height specification for concentration on water bath 8) sample removed from water bath when volume reaches ~6 mL 9) N bath temp no higher than 39 deg C	1) Add surrogate/spike directly to sample bottle 2) Add 300 - 500 mL of methylene chloride to the distilling flask of the extractor 3) CLLE for 18 - 24 hours 4) Extract dried using Na_2SO_4 in drying columns 5) Rinse the Erlenmeyer flask, which contained the solvent extract, with 20 - 30 mL of methylene chloride to complete the quantitative transfer 6) water bath temp 15-20 deg C above solvent boiling temp 7) partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-20 min 8) sample removed from water bath when volume reaches 1 mL 9) N bath temp 35 deg C
QC - Spikes	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - LCS	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

FIGURE 1

EXAMPLE OF SEMIVOLATILES LOGBOOK PAGE



SV/SIM Sep

KATAHDIN ANALYTICAL SERVICES, INC.
ORGANIC EXTRACTIONS LOG - AQUEOUS SEMI-VOLATILES

Extraction Method: (check one)	SW846 3520 (CLLE)		SW846 3510 (SEP) ✓		SW846 3535 (SPE)	
Analytical Method: (check one)	SW846 8270 ✓	EPA 625	CLP OLM04.2	CLP OLC02.1	CLP SOM01.2	
Standards	Surrogate ID (1): SV2350		Spike ID (1): SV352		Spike ID (3):	
	Surrogate ID (2): SV2349		Spike ID (2): SV2351			
Solvents/Acid/Base	Solvent Lot # (MeCl2): H40631		H_2SO_4 # 435026		NaOH Lot # H22101	
Consumables	Filter Paper Lot # K11472305		$NaSO_4$ Lot # 279069003		Vial Lot # 000 94428	
Nitrogen Water Bath Temperature 35°C			pH (1st Extraction) ≥11		pH (2nd Extraction) ≤2	
Prep Start Time: 0930	Prep End Time: 1230	CLLE Acid Start:	CLLE Acid End:	CLLE B/N Start:	CLLE B/N End:	

Date Extracted	Ext. Init.	Sample ID	Initial Vol. mL	Surr. Vol.	Spike Vol.	Fraction SV	Fraction SM	Final Vol. mL	Date Conc.	Tray Location	Initials	Comments
10/24/09	CB	WG470483-1	1000	1mL each	NR	✓	✓	1mL	10/26/09	SV492 C8	CB	Run=R112490
		WG470483-2	↓	1mL	1mL					B9		Run=R112487
		-3	980							B10		MS/SC6526-11h
		-4	1000				✓			B1		MSD ↓ -11h
		WG470484-2	1000			✓				C2		
		-3	↓	↓	↓	↓		↓	↓	C3	↓	

CB 10/26/09

Date Extracted	Ext. Init.	Sample ID	Initial Vol. mL	Surr. Vol.	Spike Vol.	Fraction SV	Fraction SM	Final Vol. mL	Date Conc.	Tray Location	Initials	Comments
10/24/09	CB	SC6526-1a	1060	1mL	NR		✓	1mL	10/26/09	SV492 C4	CB	
		-3c	1010							C5		
		-5d	1030							C6		
		-7c	1060							C7		40% dilute surr.
		-9c	1030							C8		
		-11h	1030							C9		MS/D
		-13c	1020							C10		
		-15d	1010							D1		
		-17c	1060							D2		
		-19d	↓							D3		
		-21c	↓							D4		
		-23c	1020							D5		
↓		-25c	1040				↓			D6		
		SC6525-2e	1060			✓				D7		
		-3c	↓			↓				D8		
		-4e	↓							D9		
		-5d	1040							D10		
↓	↓	-6d	1040	↓	↓	↓		↓	↓	E1	↓	

Reviewed By_____ Date_____

QAEX173 00000089

QAEX173 00000090

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

FIGURE 2

SOLVENT/REAGENT LOT CHECK LOGBOOK

SOLVENT:

LOT#:

DATE RECEIVED:

DATE CONCENTRATED:

CONCENTRATED BY:

PREP METHOD:

TRAY LOCATION:

ANALYZED BY:

PASS/FAIL:

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

FIGURE 3

LCS/MATRIX SPIKE COMPONENT LIST

BASE/NEUTRALS	
1-Methylnaphthalene	Bis (2-chloroethoxy) methane
1,1-Biphenyl	Bis (2-chloroethyl) ether
1,2,4-Trichlorobenzene	Bis (2-Chloroisopropyl) ether)
1,2-Dichlorobenzene	Bis(2-Ethylhexyl)adipate
1,3-Dichlorobenzene	Bis (2-ethylhexyl) phthalate
1,4-Dichlorobenzene	Butylbenzyl phthalate
1,4-Dioxane	Caprolactam
2,4-Dinitrotoluene	Carbazole
2,6-Dinitrotoluene	Chrysene
2-Chloronaphthalene	Dibenz (a, h) anthracene
2-Methylnaphthalene	Dibenzofuran
2-Nitroaniline	Diethyl phthalate
3,3'-Dichlorobenzidine	Diethyl adipate
3-Nitroaniline	Dimethyl phthalate
4-Bromophenylphenyl ether	Di-n-butylphthalate
4-Chloroaniline	Di-n-octyl phthalate
4-Chlorophenylphenyl ether	Fluoranthene
4-Nitroaniline	Fluorene
Acenaphthene	Hexachlorobenzene
Acenaphthylene	Hexachlorobutadiene
Acetophenone	Hexachlorocyclopentadiene
Aniline	Hexachloroethane
Anthracene	Indeno (1,2,3-cd) pyrene
Atrazine	Isophorone
Azobenzene	Naphthalene
Benzaldehyde	Nitrobenzene
Benzidine	N-Nitrosodimethylamine
Benzo (a) Anthracene	N-Nitroso-di-n-propylamine
Benzo (a) pyrene	N-Nitrosodiphenylamine
Benzo (b) fluoranthene	Phenanthrene
Benzo (ghi) perylene	p-toluidine
Benzo (k) fluoranthene	Pyrene
Benzyl alcohol	Pyridine

ACIDS		
2, 3, 4, 6-Tetrachlorophenol	2-Chlorophenol	Benzoic acid
2,4,5-Trichlorophenol	2-Methylphenol	Ethyl methanesulfonate
2,4,6-Trichlorophenol	2-Nitrophenol	Methyl methanesulfonate
2,4-Dichlorophenol	4,6-Dinitro-2-methylphenol	Pentachlorophenol
2,4-Dimethylphenol	4-Chloro-3-methylphenol	Phenol
2,4-Dinitrophenol	4-Methylphenol	
2,6-Dichlorophenol	4-Nitrophenol	

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

FIGURE 4

APPENDIX IX LCS/MATRIX SPIKE COMPONENT LIST

1,2,4,5-Tetrachlorobenzene	Hexachloropropene
1,3,5-Trinitrobenzene	Isodrin
1,4-Naphthoquinone	Isosafrole
1-Chloronaphthalene	Kepone
1-Naphthylamine	m-Dinitrobenzene
2,4-D	Methapyrilene
2-Acetyl aminofluorene	Methyl parathion
2-Naphthylamine	n-Nitrosodiethylamine
2-Picoline	n-Nitrosodi-n-butylamine
3,3-Dimethylbenzidine	n-Nitrosomethylethylamine
3-Methylcholanthrene	n-Nitrosomorpholine
4-Aminobiphenyl	n-Nitrosopyrrolidine
4-Nitroquinoline-1-oxide	n-Nitrotrosopiperidine
5-Nitro-o-toluidine	O,O,O-Triethyl phosphorothioate
7,12-Dimethylbenz(a)anthracene	o-Toluidine
a,a-Dimethylphenethylamine	Parathion
Acetophenone	p-Dimethylaminoazobenzene
Aramite	Pentachlorobenzene
Chlorobenzilate	Pentachloronitriobenzene
Diallate	Phenacetin
Dibenz(a,j)acridine	Phorate
Dimethoate	p-Phenylenediamine
Dinoseb	Pronamide
Diphenylamine	Safrole
Disulfoton	Silvex (2,4,5-TP)
Famphur	Sulfotep
Hexachlorophene	Thionazin

TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

Prepared By: George Brewer Date: 12/97

Approved By:

Group Supervisor: George Brewer Date: 02/01/01

Operations Manager: Joh C Burton Date: 2/2/01

QA Officer: Deborah J. Nadeau Date: 2.1.01

General Manager: Deborah F. Kelfer Date: 2/08/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 1311	Changed figures, inserted database references. Format changes, added pollution prevention.	DN	2.1.01	2/1/01
02 1311	Modified to reflect change from TCLP database to handwritten logbooks. Changed metals spiking instructions	LAD	030805	030805
03	Added expiration dates for TCLP fluids (1YR) Added DOC requirement Revised TCLP logbook to include SPLP and spaces for pH and exp. dates.	LAD	01/07	01/07
04	Sect. 4: Added use of fluorinated extraction vessels for organics. Updated TCLP/SPLP Logbook example.	LAD	03/08	03/08
05	Updated Figure 8 - TCLP Extraction logbook page.	LAD	03/09	03/09

TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-510-05,** titled **Toxicity Characteristic Leaching Procedure (TCLP) for Inorganic and Non-Volatile Organic Analytes**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-510-05,** titled **Toxicity Characteristic Leaching Procedure (TCLP) for Inorganic and Non-Volatile Organic Analytes**.

Recipient: _____ Date: _____

TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to define the procedures used by Katahdin Analytical Services, Inc., personnel for TCLP extraction of samples for inorganic and non-volatile organic components using USEPA Method 1311 (Test Methods for Evaluating Solid Waste, Physical / Chemical Methods, US EPA SW846), with the modifications discussed in Table 2.

The TCLP (Toxicity Characteristic Leaching Procedure) is designed to determine the mobility of both organic and inorganic analytes present in liquid, solid, and multiphasic wastes.

If a total analysis of the waste demonstrates that individual analytes are not present in the waste, or that they are present but at such low concentrations that the appropriate regulatory levels could not possibly be exceeded, the TCLP need not be run.

If an analysis of the liquid fractions of the TCLP extract indicates that a regulated compound is present at a concentration that, after accounting for dilution from the other fractions of the extract, would be equal to or above the regulatory level for that compound, then the waste is hazardous and it is not necessary to analyze the remaining fractions of the extract. The regulated toxicity characteristic analytes are listed in Table 3.

1.1 Definitions - None.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of, analysts experienced in TCLP extractions. Each analyst must demonstrate the ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, Personnel Training and Demonstration of Capability.

It is the responsibility of all Katahdin technical personnel involved in TCLP extractions to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to ensure that members of their group follow this SOP, to ensure that their work is properly documented, and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be aware of inherent laboratory hazards, proper disposal procedures for contaminated materials, and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method may not be

precisely known; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets (MSDS) is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with the Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of a respirator and all safety equipment. Each analyst shall receive a safety orientation from their Department Manager or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

Wastes from TCLP extraction may contain acids, heavy metals, toxic organics, and other toxic components and should be disposed of in a manner appropriate to the hazards they present. Further information regarding waste classification and disposal may be obtained by consulting the Katahdin Hazardous Waste Management Plan and the Department Manager.

2.0 SUMMARY OF METHOD

2.1 For liquid wastes (i.e., those containing less than 0.5% dry solid material), the waste, after filtration through a 0.6 to 0.8 µm glass fiber filter, is defined as the TCLP extract.

2.2 For wastes containing greater than or equal to 0.5% solids, the liquid phase is first separated from the solid phase and stored for later analysis. The particle size of the solid phase is reduced, if necessary, and the solid phase is extracted with an amount of extraction fluid equal to 20 times its weight. The composition of the extraction fluid employed depends on the alkalinity of the solid phase of the waste. After extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8 µm glass fiber filter.

2.3 If they are compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract and these are analyzed together. If they are incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

3.0 INTERFERENCES

Because the dissolved solids contents of TCLP extracts are typically high, analyses of these extracts are often troubled by matrix interferences. Methods to detect and overcome matrix interferences are integral to the TCLP procedure and are discussed in detail in Section 8.0, Quality Control.

Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

4.0 APPARATUS AND MATERIALS

4.1 Agitation apparatus (rotary extractor) - The agitation apparatus must be capable of rotating the extraction vessel in an end-over-end fashion at 30±2 revolutions per minute (rpm) – see Figure 1. Each of the laboratory's rotary extractors is equipped with a device that displays the actual rotation rate in rpm. The rotation rate of each extractor is monitored before each use, and the measured rotation rates are recorded in a logbook maintained for that purpose (see Figure 7). If the measured rotation rate of an extractor is outside the range 30±2 rpm, it must be taken out of service until it can be repaired.

4.2 Extraction vessels - must fit the rotary extractor and have sufficient capacity to hold the sample and the extraction fluid (jars with capacities of 2.2 L are normally used). The vessel must be made of borosilicate glass or fluorinated polyethylene if the extract is to be analyzed for organics. If the extract is to be analyzed only for inorganics, polyethylene or polypropylene containers may be used.

4.3 Filter Holder - Filter holders for pressure filtration are used. They are constructed of type 316 stainless steel (with or without PTFE linings) and are capable of sustaining internal pressures exceeding 50 psi. These devices have an internal capacity of 1.5 L and accommodate glass fiber filters 142 mm in diameter.

4.4 Filters - Borosilicate glass fiber filters containing no binder materials and having an effective pore size of 0.6 to 0.8 µm, 142 mm diameter or equivalent. Prefilters must not be used. Glass fiber filters are fragile and should be handled with care. Filters should be acid-washed with 1N HNO_3 and triple rinsed with laboratory reagent grade water (minimum 500 mL/ rinse) prior to use.

4.5 pH meter accurate to ±0.05 units at 25°C. The pH meter must be calibrated on each day of use.

4.6 pH indicator strips covering the pH range 0 - 14 in increments of 1 pH unit.

4.7 Laboratory balance accurate to within ±0.01 grams (all weight measurements are to be within ±0.1 grams).

4.8 Beakers flasks, glass, 500 mL..

4.9 Watch glasses, appropriate diameter to cover beakers.

4.10 Magnetic stirrer.

5.0 REAGENTS

Reagent grade chemicals shall be used in all tests. Other grades may be used only if it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.1 Laboratory reagent grade water – Water free of any analyte of interest. Laboratory reagent grade water should be monitored periodically for impurities.

5.2 Hydrochloric acid, concentrated (HCl) – reagent grade.

5.3 Nitric acid, concentrated (HNO_3) – reagent grade.

5.4 Hydrochloric acid, 1N. Dilute 83 mL reagent grade HCl to 1000 mL with laboratory reagent grade water.

5.5 Nitric acid, 1N, for acid-washing filters. Dilute 63 mL reagent grade HCl to 1000 mL with laboratory reagent grade water.

5.6 Sodium hydroxide (NaOH) – reagent grade, pellets.

5.7 Glacial acetic acid (CH_3COOH) – reagent grade.

5.8 Extraction Fluid #1 - Add 114 mL glacial acetic acid and 51.4 g sodium hydroxide to approximately 1500 mL of laboratory reagent grade water in a clean borosilicate glass extraction vessel reserved for this purpose. Shake until the sodium hydroxide is completely dissolved. Pour this solution into a clean, graduated 20 L carboy reserved for Extraction Fluid #1 and rinse the extraction vessel three times with approximate liter volumes of laboratory reagent grade water, adding the rinsates to the carboy. Add laboratory reagent grade water to the carboy to bring the volume to the 20 L graduation. Cap the carboy and agitate until the fluid is well mixed. When correctly prepared, the pH of this fluid will be 4.93 ±0.05. The fluid may be used for up to one year from the preparation date.

5.9 Extraction Fluid #2 - Add approximately 10 L of laboratory reagent grade water to a graduated 20 L carboy reserved for Extraction Fluid #2. Add 114 mL glacial acetic

acid to the carboy, and then add laboratory reagent grade water to bring the volume to the 20 L graduation. Cap the carboy and agitate until the fluid is well mixed. When correctly prepared, the pH of this fluid will be 2.88 ±0.05. The fluid may be used for up to one year from the preparation date.

NOTE: The pH of each extraction fluid must be checked prior to each use to ensure that it has been prepared accurately, and the measured pH is recorded in the Non-Volatile TCLP Extraction Logbook (Figure 8) for each sample extracted. Details of the preparation of these fluids (reagent lot numbers, volumes, and masses; measured pH; etc.) are recorded in the TCLP Fluid Preparation and Use Logbook (Figure 6). Upon preparation, each new batch of extraction fluid is assigned a 3-digit batch number by the analyst (batches are numbered consecutively), and the Katahdin Sample Number of each client sample extracted with a particular fluid batch is recorded in the TCLP Fluid Preparation and Use Logbook. Extraction fluids are monitored for impurities as described in Section 8.0 of this SOP.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

All samples shall be collected in a soil jar using an appropriate sampling plan.

6.1 Sufficient sample must be collected to support the preliminary determinations and to provide an extract volume adequate for all analytical and quality control purposes. The necessary sample size will depend on the solids content of the waste, but in no instance should less than 250 g of waste be provided to the laboratory.

6.2 Preservatives shall not be added to samples before extraction. Samples should be stored at 4°C and opened immediately prior to TCLP extraction.

6.3 TCLP extracts should be prepared for analyses and analyzed as soon as possible following TCLP extraction. Extracts for metals analysis must be acidified to a pH < 2 with nitric acid. Extracts for other analyses should be preserved according to the guidance given in the individual analytical methods. Extracts for organic analyte determinations shall not be allowed to come into contact with the atmosphere (i.e., no headspace) to prevent losses.

6.4 Sample holding times for non-volatile TCLP extraction and analysis summarized in the following table:

TCLP PARAMETER	FROM COLLECTION TO TCLP EXTRACTION	FROM TCLP EXTRACTION TO PREPARATIVE EXT'N	FROM PREP EXT'N TO ANALYSIS
PEST/HERBS	14	7	40

SEMIVOLATILES	14	7	40
MERCURY	28	N/A	28
METALS EXCEPT MERCURY	180	N/A	180

7.0 PROCEDURES

The procedure consists of a series of preliminary evaluations of the waste, followed by the actual extraction. Flow charts summarizing the procedure appear as Figures 2 and 3. Preliminary evaluations are to be performed on a minimum 100 g aliquot of the waste. This aliquot may not actually undergo TCLP extraction. These preliminary evaluations include: (1) determination of the percent solids, Section 7.1; (2) determination of whether the waste contains insignificant solids and is, therefore, its own extract after filtration, Section 7.2; (3) particle size evaluation, Section 7.3; and (4) determination of the appropriate extraction fluid to be used for the TCLP extraction, Section 7.4.

All information and measurements pertaining to TCLP extractions are recorded in the Non-Volatile TCLP Extraction Logbook (Figure 8). In the following procedure, the section or line of the Non-Volatile TCLP Extraction Logbook page in which the pertinent information should be recorded is indicated in bold, e.g. **Section II** or **Line C**.

PRELIMINARY EVALUATIONS

7.1 Determination of Percent Solids (**Section I**) - Percent solids is defined for TCLP as that fraction of a waste sample (as a percentage of the total sample) from which no liquid may be forced out by an applied pressure, as described below.

If the waste will obviously yield no liquid when subjected to pressure filtration (i.e., is 100% solids) the percent solids determination may be omitted. Proceed to Section 7.3, Particle Size Evaluation.
If the sample is liquid or multiphasic, liquid/solid separation by filtration is required to make a preliminary determination of percent solids. This involves the filtration device. The procedure is as follows, Sections 7.1.1 through 7.1.9:

7.1.1 Pre-weigh the filter (**Line A**) and the container that will receive the filtrate (filtrate vessel) (**Line B**).

7.1.2 Assemble the filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure.

7.1.3 Weigh out a subsample of the waste (100 gram minimum) and record the combined weight of the weigh boat and waste (**Line C**).

7.1.4 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged, prior to filtration. Centrifugation is to be used only as an aid to filtration. If centrifugation is used, the liquid should be decanted and filtered followed by filtration of the solid portion of the waste through the same filtration system.

7.1.5 Quantitatively transfer the waste sample (liquid and solid phases) to the filter holder, spreading the waste sample evenly over the surface of the filter. If filtration of the waste at 4°C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering.

7.1.6 Weigh the weigh boat and any residue clinging to it (**Line D**). Determine the total weight of waste to be filtered by subtracting the weight of the weigh boat and residue from the weight of the weigh boat and waste (**Line E**).

7.1.7 Gradually apply vacuum or gentle pressure of 1-10 psi until air or pressurizing gas moves through the filter, collecting any filtrate in the pre-weighed filtrate vessel. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any 2-minute interval slowly increase the pressure in 10-psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2-minute interval, proceed to the next 10-psi increment. When the pressurizing gas begins to move through the filter, or when liquid flow has ceased at 50 psi (i.e., filtration does not result in any additional filtrate within any 2 minute period), stop the filtration.

The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase.

7.1.8 Weigh the filtrate vessel and its contents (**Line F**). Determine the weight of the liquid phase by subtracting the weight of the filtrate vessel from the total weight of the filtrate-filled container (**Line G**).

7.1.9 Calculate the percent wet solids as follows (**Line H**):

$$\text{Percent wet solids} = \frac{\text{(Total weight of waste)-(Weight of liquid phase)}}{\text{Total weight of waste}}$$

7.2 If the percent solids determined in Section 7.1.9 above is equal to or greater than 0.5% and the weight of water entrained in the filter is small in comparison with the weight of the solid phase, then proceed to Section 7.3 to determine whether the solid material requires particle size reduction. Continue with Section 7.2 if it is noticed that the amount of the filtrate entrained in wetting the filter is significant in proportion to the

weight of the solid phase. If the percent solids determined in Section 7.1.9 is less than 0.5%, then proceed to Section 7.5.4 using a fresh portion of the waste.

7.2.1 Remove the solid phase and filter from the filtration apparatus.

7.2.2 Dry the filter and solid phase at 100± 20°C until two successive weighings yield the same value within ±1%. Record the weight of the filter and dry solids (**Line I**).

NOTE: Caution should be taken to ensure that the subject solid will not flash upon heating. It is recommended that the drying oven be vented to a hood or other appropriate device.

7.2.3 Calculate the weight of dry solids by subtracting the weight of the filter from the weight of the filter and dry solids (**Line J**).

7.2.4 Calculate the percent dry solids as follows (**Line L**):

$$\text{Percent dry solids} = \frac{\text{Weight of dry solids}}{\text{Total weight of waste}} \times 100$$

Note: Non-aqueous liquid samples (e.g. oils) may be entrained in the filter, and may remain in the filter after drying, contributing weight to the dried filter. If this is the case, the surface of the filter should be examined for apparent solids or particulate material. If none are found, a comment to that effect should be made in the Comments section of the Non-Volatile TCLP Extraction Logbook (e.g. "No apparent solids present – dry solid weight is due to entrained non-volatile liquid"), and the sample should be treated as if it contains less than 0.5% dry solids.

7.2.5 If the percent dry solids is less than 0.5%, then proceed to Section 7.5.4. If the percent dry solids is greater than or equal to 0.5%, proceed to Section 7.3.

7.3 Particle Size Evaluation - Visually evaluate the particle size of the solid phase of the waste. Filamentous material (cloth, paper, etc.) will require particle size reduction if it has a surface area per gram of less than 3.1 cm^3. Other solid materials require particle size reduction if the particles are greater than 1 cm in their narrowest dimension (i.e. if they will not pass through a 9.5 mm standard sieve). Particle size reduction may be accomplished by cutting, crushing, or grinding the waste to a surface area or particle size as described above. Perform particle size reduction on the solid material that will actually undergo extraction, not on that used for the preliminary determinations.

7.4 Determination of Appropriate Extraction Fluid - If the solid content of the waste is greater than or equal to 0.5%, determine the appropriate fluid for the non-volatiles extraction as follows:

7.4.1 Weigh out a small subsample of the solid phase of the waste, reduce the particle size (if necessary) to approximately 1 mm in diameter or less, and transfer 5.0 grams of the solid phase of the waste to a 500 mL beaker or Erlenmeyer flask.

7.4.2 Add 96.5 mL of laboratory reagent grade water to the beaker, cover with a watch glass, and stir vigorously for 5 minutes using a magnetic stirrer. Measure and record the pH (**Section II**). If the pH is <5.0, use Extraction Fluid #1 and proceed with the TCLP extraction, Section 7.5.

NOTE: pH measurements may initially be performed using a pH indicator strip. If the measured pH is less than 3 or greater than 7, record the pH obtained from the indicator strip to the nearest whole pH unit. If the measured pH is between 3 and 7, use the pH meter to obtain a more accurate reading and record the pH to at least one decimal place.

7.4.3 If the pH from Section 7.4.2 is >5.0, add 3.5 mL 1N HCl, stir briefly, cover with a watch glass, heat to 50°C, and hold at 50°C for 10 minutes.

7.4.4 Let the solution cool to room temperature and record the pH (**Section II**). If the pH is <5.0, use Extraction Fluid #1. If the pH is still >5.0, use Extraction Fluid #2. Proceed to the TCLP extraction, Section 7.5.

TCLP EXTRACTION FOR NON-VOLATILES

7.5 A minimum sample size of 100 grams (solid and liquid phases) is recommended. In some cases, a larger sample size may be appropriate, depending on the solids content of the waste sample, whether the initial liquid phase of the waste will be miscible with the aqueous extract of the solid, and whether inorganics, semivolatile organics, pesticides, and herbicides are all analytes of concern. Enough solids should be generated for extraction such that the volume of TCLP extract will be sufficient to perform all of the required analyses. If necessary, multiple extractions may be performed and the extracts combined and aliquoted for analysis.

7.5.1 If the waste will obviously yield no liquid when subjected to pressure filtration (i.e., is 100% solid), weigh out a subsample of the waste (100 g minimum), record the weight (**Section III**), and proceed to Section 7.5.11. If the sample is liquid or multiphasic, liquid/solid separation is required - proceed to Section 7.5.2.

7.5.2 Pre-weigh the container that will receive the filtrate (filtrate vessel) (**Line M**).

7.5.3 Assemble the filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure. Acid-wash the filter if extracting for metals components. Acid-washed filters may be used for non-volatile extractions even when metals are not of concern.

7.5.4 Weigh out a subsample of the waste (100 gram minimum) and record the combined weight of the waste and weigh boat (**Line N**). If the waste contains <0.5% dry solids, the liquid portion of the waste, after filtration, is defined as the TCLP extract. Therefore, enough of the sample should be filtered so that the amount of filtered liquid will support all of the required analyses. For wastes containing >0.5% dry solids, information is obtained in Section 7.1 to determine the optimum sample size (100 gram minimum) for filtration. Enough solids should be generated by filtration to support the analyses to be performed on the TCLP extract.

7.5.5 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration. Use centrifugation only as an aid to filtration. If centrifugation is used, the liquid should be decanted and filtered followed by filtration of the solid portion of the waste through the sample filtration system.

7.5.6 Quantitatively transfer the waste sample (liquid and solid phases) to the filter holder. Spread the waste sample evenly over the surface of the filter. If filtration of the waste at 4°C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering.

7.5.7 Weigh the weigh boat and any residue clinging to it (**Line O**). Determine the total weight of waste to be filtered by subtracting the weight of the weigh boat and residue from the weight of the weigh boat and waste (**Line P**).

7.5.8. Gradually apply vacuum or gentle pressure of 1-10 psi until air or pressurizing as moves through the filter, collecting any filtrate in the pre-weighed filtrate vessel. If this point is reached under 10 psi, and if no additional liquid has passed through he filter in any 2-minute interval, slowly increase in pressure in 10-psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2-minute interval, proceed to the next 10-psi increment. When the pressurizing gas begins to move through the filter, or when the liquid flow has creased at 50 psi (i.e., filtration does not result in any additional filtrate within a 2 minute period), stop the filtration.

The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase.

7.5.9 Weigh the filtrate vessel and its contents (**Line Q**). Determine the weight of the liquid phase by subtracting the weight of the filtrate vessel from the total weight of the filtrate-filled container (**Line R**). Decant the liquid phase into a graduated cylinder and measure and record its volume (**Line S**). Pour the liquid phase back into the filtrate vessel for storage. The liquid phase may now either be analyzed or stored at 4°C until time of analysis.

NOTE: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying pressure filtration, as outlined in Section 7.5.8, this material may not filter. If this is the case, the material within the filtration device is defined as a solid and is carried through the extraction as a solid. Do not replace the original filter with a fresh filter under any circumstances. Use only one filter.

7.5.10 Calculate the weight of wet solids by subtracting the weight of the liquid phase from the total weight of waste (**Line T**).

7.5.11 If necessary, prepare the solid portion of the waste for extraction by crushing, cutting, or grinding the waste to a surface area or particle size as described in Section 7.3. Describe the particle size reduction process in **Section IV** of the logbook. When the surface area or particle size has been appropriately altered, quantitatively transfer the solid material into an extractor bottle. Include the filter used to separate the initial liquid from the solid phase.

7.5.12 Determine the amount of extraction fluid to add to the extractor vessel as follows:

$$\text{Weight of extraction fluid} = \frac{(20)\,(\text{Weight of wet solids})}{100}$$

Slowly add this amount of appropriate extraction fluid to the extractor vessel. Record the fluid batch ID, the amount used, and the pH (measured on day of use) in **Section III** of the logbook. Close the extractor bottle tightly (Teflon tape may be used to ensure a tight seal), secure in rotary agitation device, and rotate at 30± 2 RPM during the extraction period of 18 $\pm$ 2 hours. Record the extraction start and end times and the room temperatures in **Section IV** of the logbook.

NOTE: As agitation continues, pressure may build within the extractor bottle for some types of wastes (e.g., limed or calcium carbonate containing waste may evolve gases such as carbon dioxide). To relieve excess pressure, the extractor bottle may be periodically opened (e.g., after 15 minutes, 30 minutes, and 1 hour) and vented into a hood.

7.5.13 Following the extraction, separate the contents of the vessel into its component liquid and solid phases by filtering through a new acid-washed glass fiber filter, as outlined in Section 7.5.6. For final filtration of the TCLP extract, the glass fiber filter may be changed, if necessary, to facilitate filtration.

NOTE: If the waste contained no initial liquid phase, it is only necessary to filter enough extract to support the required analyses. However, if the waste contained an initial liquid phase, the entire contents of the extraction vessel must be filtered.

7.5.14 Prepare the TCLP extract as follows:

7.5.14.1 If the waste contained no initial liquid phase, the filtered liquid material obtained from Section 7.5.13 is defined as the TCLP extract. Proceed to Section 7.5.15.

7.5.14.2 If compatible (e.g., multiple phases will not result on combination), combine the filtered liquid resulting from Section 7.5.13 with the initial liquid phase of the waste obtained in Section 7.5.8. This combined liquid is defined as the TCLP extract. Proceed to Section 7.5.15.

7.5.14.3 If the initial liquid phase of the waste, as obtained from Section 7.5.8, is not or may not be compatible with the filtered liquid resulting from Section 7.5.13, do not combine these liquids. Measure the volume of filtrate obtained in Section 7.5.13 and record in **Section IV** of the logbook. Individually analyze these two liquids, collectively defined as the TCLP extract, and combine the results mathematically, as described in Section 7.6.

7.5.15 Following collection of the TCLP extract, the pH of the extract should be measured and recorded (**Section IV**). Immediately aliquot and preserve the extract for analysis. Metals aliquots must be acidified with nitric acid to pH <2. All other aliquots must be stored under refrigeration (4°C) until analyzed.

7.6 The TCLP extract shall be prepared and analyzed according to appropriate analytical methods. TCLP extracts to be analyzed for metals shall be acid digested except in those instances where digestion causes loss of metallic analytes. If an analysis of the undigested extract shows that the concentration of any regulated metallic analyte exceeds the regulatory level, then the waste is hazardous and digestion of the extract is not necessary. However, data on undigested extracts alone cannot be used to demonstrate that the waste is not hazardous. If the individual phases are to be analyzed separately, determine the volume of the individual phases (to ± 0.5%),

conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$\text{Final Analyte Concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

where: V_1 = The volume of the first phase (L).
C_1 = The concentration of the analyte of concern in the first phase (mg/L).
V_2 = The volume of the second phase (L).
C_2 = The concentration of the analyte of concern in the second phase (mg/L).

7.6 Compare the analyte concentrations in the TCLP extract with the levels identified in the appropriate regulations. Refer to Section 8.0 for quality control requirements.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 1311 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are listed in Table 1 and are described below. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The Department Manager, Operations Manager and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed

8.1 A minimum of one method blank for every 20 extractions performed using a particular batch of extraction fluid and per 20 extractions performed in a particular extraction vessel must be extracted and analyzed for the same contaminants as all associated

samples. The method blanks are analyzed to check for laboratory contamination. A count of extractions performed in each extraction vessel is maintained in order to monitor the frequency of method blanks (1 per 20 extractions per vessel) required for each extraction vessel. .

8.1.1 After TCLP extraction, TCLP method blanks must undergo preparative extraction and analysis within method holding times (refer to Section 6.4). For this reason it may be necessary to extract more than one method blank using a particular batch of extraction fluid. For example, suppose that a sample requiring analysis for TCLP metals and semivolatiles is extracted using freshly prepared fluid from Batch 300. Because the fluid is new, a method blank is extracted with the sample and analyzed for the same components as the sample. Eight days later, a different sample requiring full TCLP analysis (metals, semivolatiles, pesticides, and herbicides) is extracted using fluid from Batch 300. Because the holding time for the previous TCLP method blank for pesticides and herbicides has expired, a new TCLP method blank must be extracted and analyzed for pesticides and herbicides. The new method blank need not be analyzed for metals and semivolatiles, because the first method blank that was prepared with fluid from Batch 300 has already been analyzed for these constituents.

8.1.2 Each TCLP method blank is identified in the TCLP extraction logbooks by a seven-character code. The first three characters are “PBT”, which stands for “Preparation Blank - TCLP”. Characters 4 through 6 consist of the three-digit preparation number of the extraction fluid. The seventh character is a letter, starting with “A” and proceeding alphabetically, which is unique to the extraction date for a particular batch of fluid. For example, “PBT316A” refers to the first TCLP method blank extracted using fluid from Batch 316; “PBT316B” refers to the second TCLP method blank extracted using the same fluid. The extraction date of each TCLP method blank is recorded in the TCLP Fluid Preparation and Use Logbook.

8.2 The laboratory recommends that a matrix spike be performed for each waste type (e.g., wastewater treatment sludge, contaminated soil, etc.) unless the result exceeds the regulatory level and the data are being used solely to demonstrate that the waste property exceeds the regulatory level. Because the laboratory charges for the preparation and analysis of TCLP matrix spikes, selection of samples for TCLP matrix spiking is left to the discretion of the client. A minimum of one TCLP matrix spike must be analyzed for each batch of 20 TCLP extractions. As a minimum, follow the matrix spike addition guidance provided in each analytical method. Additional matrix spiking directions and guidance are provided in Table 4 and Figures 4 and 5.

8.2.1 Matrix spikes are to be added after filtration of the TCLP extract and before any preservation. Matrix spikes should not be added prior to TCLP extraction of the sample.

8.2.2 Instructions for preparing TCLP matrix spikes for metals analysis are contained in Table 4. Instructions for preparing TCLP matrix spikes for organics analyses are contained in Figures 4 and 5. In order to avoid differences in matrix effects, the matrix spikes must be added to the same nominal volume of TCLP extract as that which was analyzed for the unspiked sample.

8.2.3 Matrix spike recoveries are calculated by the following formula:

$$\text{Recovery (\%)} = 100\ (X_s - X_u)/K$$

where: X_s = measured value for the spiked sample,
X_u = measured value for the unspiked sample, and
K = known value of the spike in the sample

8.2.4 The purpose of the matrix spike is to monitor the performance of the sample preparation and analytical methods used and to determine whether matrix interferences exist. Use of internal calibration methods (e.g. the method of standard additions [MSA]), modification of the analytical methods, or use of alternate analytical methods may be needed to accurately measure the analyte concentration of the TCLP extract when the recovery of the matrix spike is below the expected analytical method performance. Metallic analytes must be quantitated by the method of standard additions if the TCLP matrix spike recovery for the analyte is less than 50% and the measured concentration of the analyte in the unspiked aliquot is within 20% of the regulatory level.

8.3 Each new analyst must demonstrate her/his ability to perform the method acceptably by while being witnessed by an analyst who is experience in performing the method. To successfully demonstrate the method, the analyst must perform the method in conformance with all the requirements of the SOP, referring to the SOP for guidance as necessary. In addition, each analyst must demonstrate the ability to produce TCLP Extraction Blanks that are free of contamination. This demonstration will require the analyst to collect and file the analytical results from four Extraction Blanks that he/she has generated.

8.4 All quality control measures described in the appropriate analytical methods shall be followed.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Department Manager and with the QAO.

Refer to the current revisions of USEPA Method 1311 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, US EPA SW846, Third Edition, Final Update I (7/92), Method 1311

Federal Register, Volume 55, Number 126, Friday, June 29, 1990, PP 26986-26998

Federal Register, Volume 57, Number 227, Tuesday, November 24, 1992, PP 55114-55117

List of Tables and Figures

TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

TABLE 1

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Toxicity Characteristic Leaching Procedure (TCLP)/ EPA 1311	Method Blanks	One per 20 samples extracted using a particular batch of extraction fluid.	Refer to individual analytical methods.	Prepare fresh extraction fluid and repeat TCLP extraction of all associated samples.
		One per 20 samples extracted in a particular extraction vessel.	Refer to individual analytical methods.	Remove extraction vessel from service.
	Matrix Spike	One per 20 TCLP extractions performed (required). One per waste type (suggested, left to discretion of client).	For metallic analytes, >50% if native analyte concentration is within ± 20% of regulatory level. For other analytes, refer to appropriate analytical methods.	For metallic analytes, quantitate by method of standard additions. For other analytes, refer to appropriate analytical methods.
	Demonstration of analyst proficiency; accuracy and precision	One time demonstration by each analyst performing the method	New analyst's performance of the method is witnessed by an experienced analyst. New analyst must produce method blanks that meet all method and laboratory acceptance criteria.	Repeat analysis until able to demonstrate acceptable performance of the method to witnessing analyst and by producing acceptable method blanks; document successful performance in personal training file.

TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-510-05	EPA METHOD 1311
Reagents	Extraction Fluid #1 prepared using sodium hydroxide pellets.	Extraction Fluid #1 prepared using 1N sodium hydroxide solution.
QC - Method Blanks	Frequency of one method blank per 20 extractions performed using a particular batch of extraction fluid and per 20 extractions performed in a particular extraction vessel.	Frequency of one method blank per 20 extractions performed in a particular extraction vessel.
QC - Spikes	Matrix spike **recommended** for each waste type.	Matrix spike **required** for each waste type.

TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

TABLE 3

TOXICITY CHARACTERISTIC CONSTITUENTS AND REGULATORY LEVELS

Constituent	Regulatory Level (mg/L)
Arsenic	5.0
Barium	100.0
Benzene	0.5
Cadmium	1.0
Carbon tetrachloride	0.5
Chlordane	0.03
Chlorobenzene	100.0
Chloroform	6.0
Chromium	5.0
o-Cresol	200.0
m-Cresol	200.0
p-Cresol	200.0
Cresol	200.0
2,4-D	10.0
1,4-Dichlorobenzene	7.5
1,2-Dichloroethane	0.5
1,1-Dichloroethylene	0.7
2,4-Dinitrotoluene	0.13
Endrin	0.02
Heptachlor (and its hydroxide)	0.008
Hexachlorobenzene	0.13
Hexachloro-1,3-butadiene	0.5
Hexachloroethane	3.0
Lead	5.0
Lindane	0.4
Mercury	0.2
Methoxychlor	10.0
Methyl ethyl ketone	200.0
Nitrobenzene	2.0
Pentachlorophenol	100.0
Pyridine	5.0
Selenium	1.0
Silver	5.0
Tetrachloroethene	0.7
Toxaphene	0.5
Trichloroethylene	0.5
2,4,5-Trichlorophenol	400.0
2,4,6- Trichlorophenol	2.0
2,4,5-TP (Silvex)	1.0
Vinyl Chloride	0.2

TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

TABLE 4

TCLP MATRIX SPIKING FOR METALLIC ANALYTES

SPIKING INSTRUCTIONS			
Sample or Solution Name	**Component Solution Name**	**Source of Component**	**Amount of Component Added per 50 mL Final Volume (mL)**
TCLP Matrix Spike (ICP)	CLPP-SPK-1	Inorganic Ventures	0.050
	CLPP-SPK-INT1	Lab Prepared (see below)	0.50
TCLP Matrix Spike (Mercury)	1000 ug/L Hg Standard	Prepared from 1000 mg/L stock standard	0.10

Note: Spiking must be performed after TCLP extraction and before preservation.

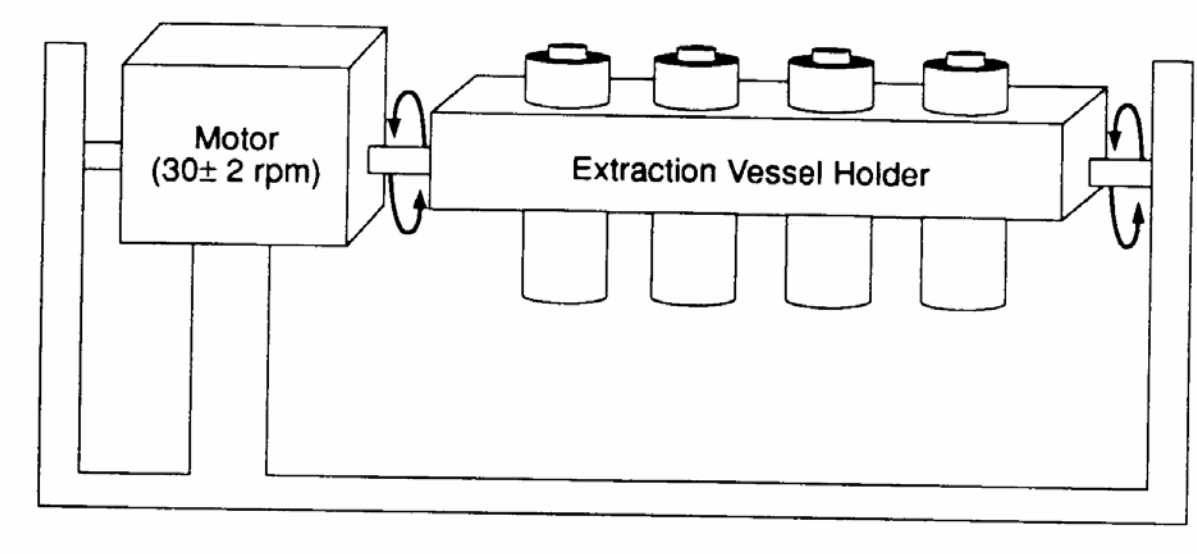
PREPARATION OF INTERMEDIATE SPIKING SOLUTIONS			
Solution Name	**Component Solution Name**	**Source of Component**	**Amount of Component Added per 100 mL Final Volume (mL)**
CLPP-SPK-INT1	QCP-CICV-3	Inorganic Ventures	10.0
	1000 mg/L Sb	High Purity Standards	5.0
	10000 mg/L K	High Purity Standards	10.0
	10000 mg/L Na	High Purity Standards	7.5
	10000 mg/L Mg	High Purity Standards	5.0
	10000 mg/L Ca	High Purity Standards	2.5
1000 ug/L Hg Standard	1000 mg/L Hg	Inorganic Ventures	0.10

ELEMENT CONCENTRATIONS IN MATRIX SPIKES AND SPIKING SOLUTIONS				
Element	**CONCENTRATION IN SOLUTION, mg/L**			
	TCLP Matrix Spike	**CLPP-SPK-1**	**CLPP-SPK-INT1**	**1000 ug/L Hg Std.**
Arsenic	2.000		200	
Barium	2.000	2000		
Cadmium	0.050		5	
Chromium	0.200	200		
Lead	0.500		50	
Selenium	2.000		200	
Silver	0.050	50		
Mercury	0.0020			1000

TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

FIGURE 1

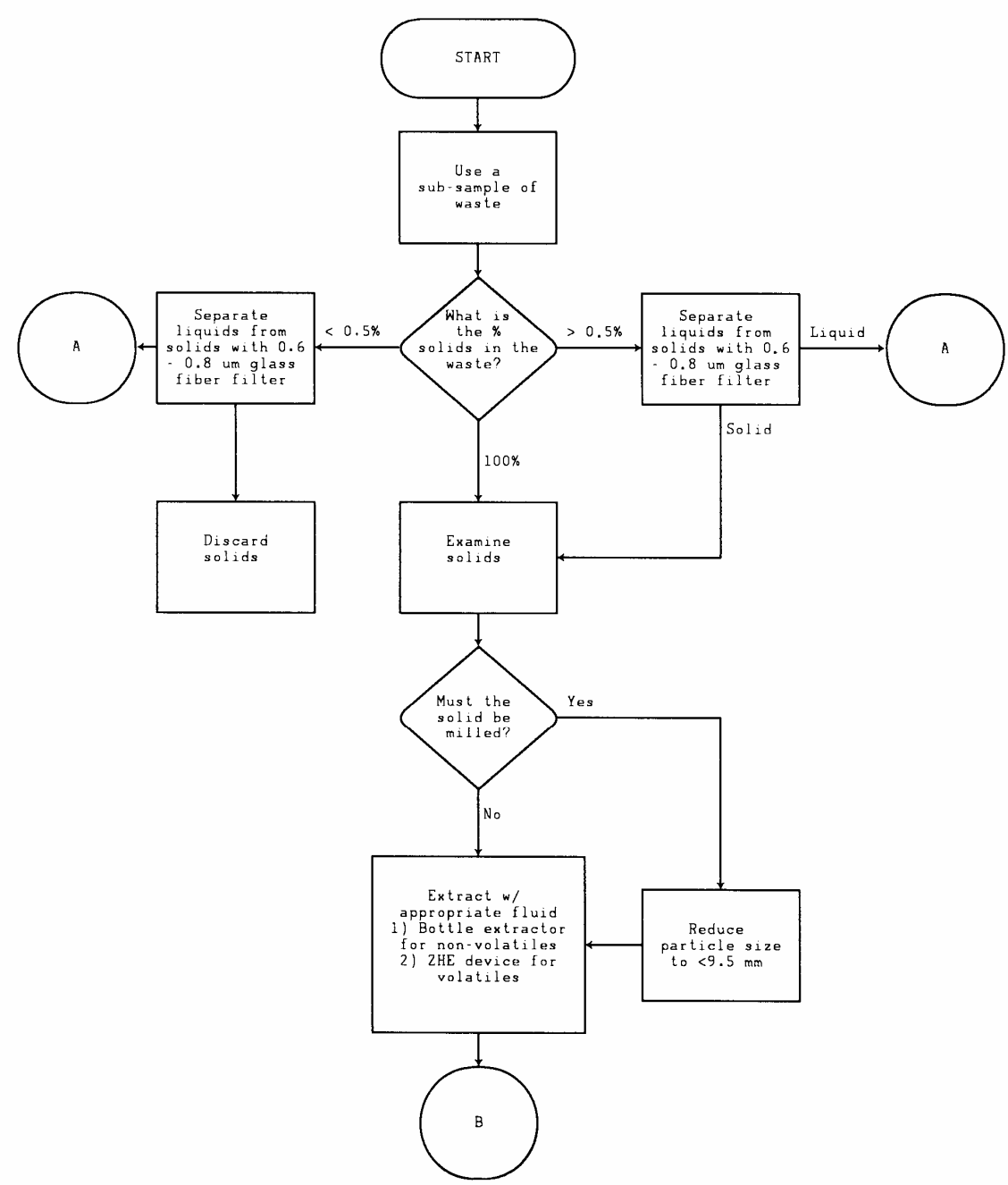
ROTARY AGITATION APPARATUS



TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

FIGURE 2

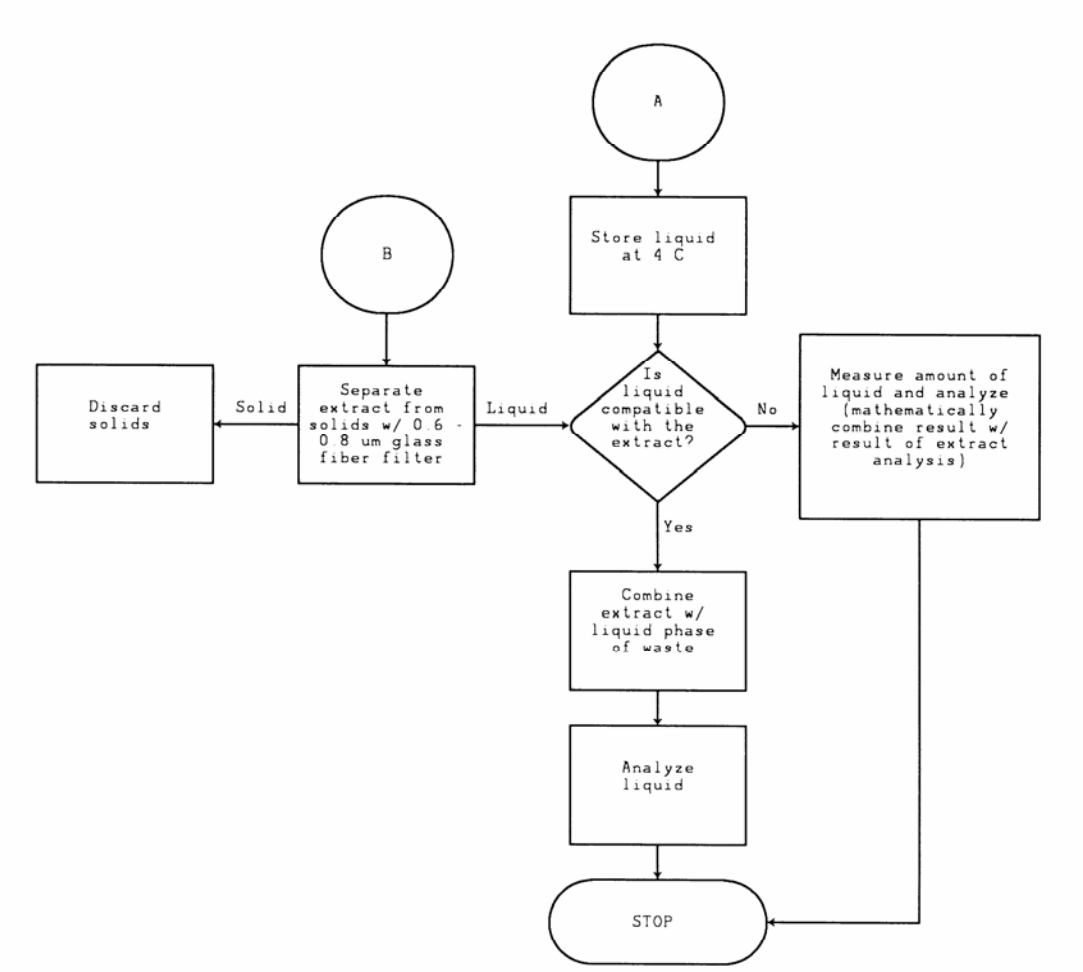
TCLP FLOW CHARTS



TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

FIGURE 3

TCLP FLOW CHARTS



TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

FIGURE 4

SVOA TCLP MATRIX SPIKE AND SURROGATE GUIDELINES

MATRIX SPIKE

The following compounds are reported for TCLP matrix spikes, although a full list spike solution is utilized (refer to SOP CA-502, current revision). Acid extractable compounds are at 100 ug/mL and base/neutral extractable compounds are at 50 ug/mL. 1.0 mL of this mix is added to the sample designated for the TCLP matrix spike.

Pyridine
1,4-Dichlorobenzene
2-Methylphenol
3-,4-Methylphenol*
Hexachloroethane
Nitrobenzene
Hexachlorobutadiene
2,4,6-Trichlorophenol
2,4,5-Trichlorphenol
2,4-Dinitrotoluene
Hexachlorobenzene
Pentachlorophenol

* Due to coelution on the GC/MS, 3-methylphenol and 4-methylphenol are reported as the combined concentration for the two isomers; the matrix spike solution contains 4-methylphenol at 100 ug/mL.

SURROGATE

The following surrogate compounds are reported for TCLP samples, although the surrogate mix also includes one additional surrogate (refer to SOP CA-502, current revision). Acid extractable surrogates are at 100 ug/mL and base/neutral extractable surrogates are at 50 ug/mL. 1.0 mL of this mix is added to all samples.

2-Fluorophenol	100 ug/mL
Phenol-d5	100 ug/mL
Nitrobenzene-d5	50 ug/mL
2-Fluorobiphenyl	50 ug/mL
2,4,6-Tribromophenol	100 ug/mL
Terphenyl-d14	50 ug/mL

TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

FIGURE 5

PESTICIDE TCLP MATRIX SPIKE AND SURROGATE GUIDELINES

MATRIX SPIKE

The following compounds are reported for TCLP matrix spikes, although a full list spike solution is utilized (refer to SOP CA-515, current revision). All compounds are at 0.5 ug/mL. 1.0 mL of this mix is added to the sample designated for the TCLP matrix spike.

Endrin
Heptachlor
Methoxychlor
Lindane
Heptachlor Epoxide

SURROGATE

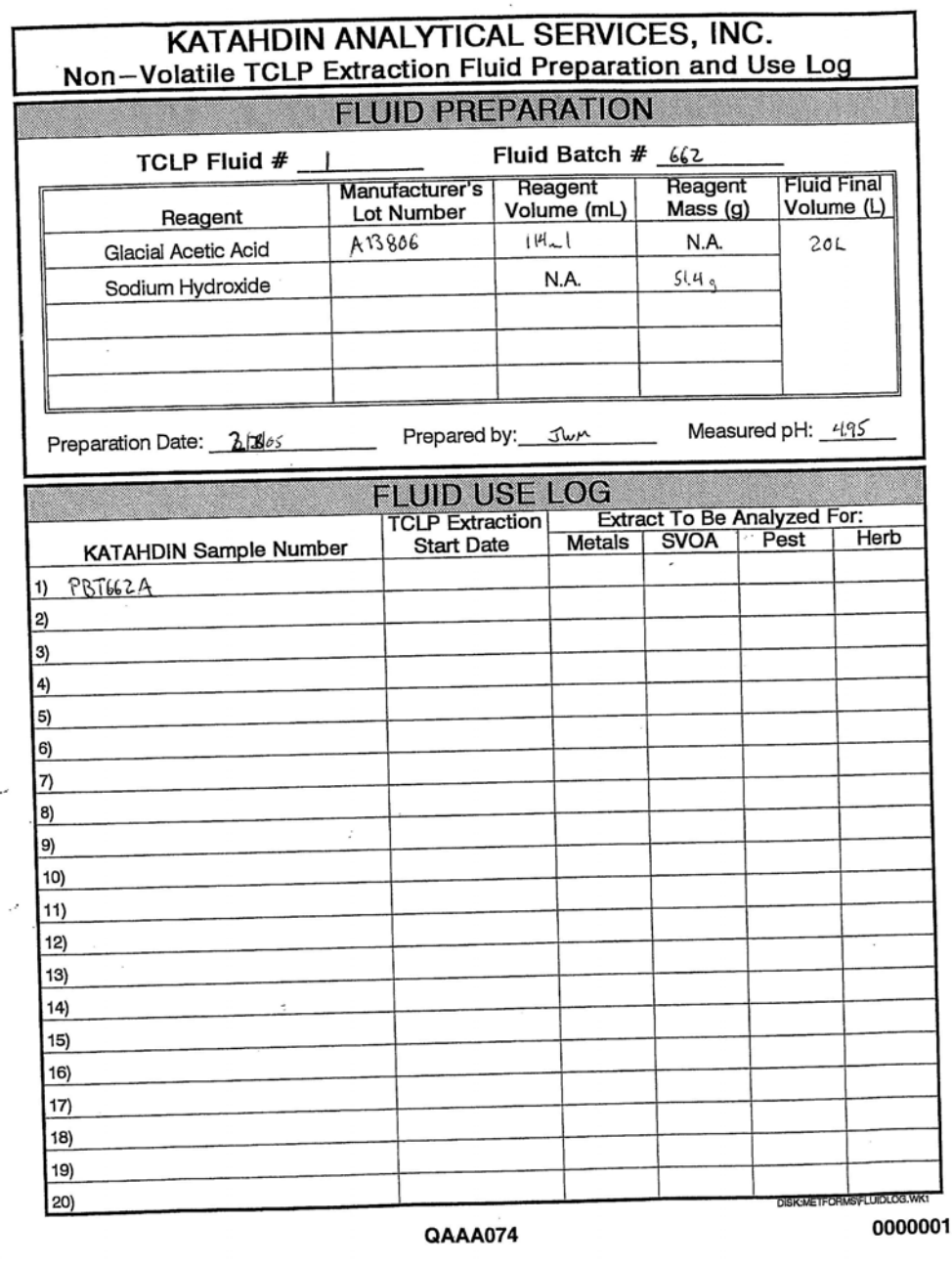
Surrogates are at 1.0 ug/mL. 1.0 mL of this mix is added to all samples.

Decachlorobiphenyl (DCB)
Tetrachloro-m-xylene (TCMX)

TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

FIGURE 6

EXAMPLE PAGE FROM TCLP FLUID USE LOGBOOK



KATAHDIN ANALYTICAL SERVICES, INC.
Non–Volatile TCLP Extraction Fluid Preparation and Use Log

FLUID PREPARATION

TCLP Fluid # 1 Fluid Batch # 662

Reagent	Manufacturer's Lot Number	Reagent Volume (mL)	Reagent Mass (g)	Fluid Final Volume (L)
Glacial Acetic Acid	A13806	1141	N.A.	20L
Sodium Hydroxide		N.A.	51.4 g	

Preparation Date: 2/28/05 Prepared by: JWM Measured pH: 4.95

FLUID USE LOG

KATAHDIN Sample Number	TCLP Extraction Start Date	Extract To Be Analyzed For: Metals	SVOA	Pest	Herb
1) PBT662A					
2)					
3)					
4)					
5)					
6)					
7)					
8)					
9)					
10)					
11)					
12)					
13)					
14)					
15)					
16)					
17)					
18)					
19)					
20)					

DISK:\METFORMS\FLUIDLOG.WK1

QAAA074

0000001

TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

FIGURE 7

EXAMPLE PAGE FROM ROTARY EXTRACTOR RPM VERIFICATION LOGBOOK

KATAHDIN ANALYTICAL SERVICES, INC.

ROTARY EXTRACTOR RPM VERIFICATION LOGBOOK

EXTRACTOR #1: TOP SHELF SERIAL NUMBER: NONE
EXTRACTOR #2: MIDDLE SHELF SERIAL NUMBER: 1173
EXTRACTOR #3: BOTTOM SHELF SERIAL NUMBER: 1169

Please record the number of RPMs for each extractor each time they are used.

Date	Initials	Extractor #1	Extractor #2	Extractor #3	Comments
10/06/06	GRB	out of service	30	30	Replaced fuse in Ext. #2
10/	ALL	out of service	29	Noise	
11/01/06	DMF	↓	30	↓	
11/30/06	DMF	↓	not in use	30	
12/27/06	DMF	↓	↓	30	
01/09/07	DMF	↓	↓	30	
01/11/07	DMF	↓	↓	30	
01/22/07	DMF	↓	↓	29	
01/25/07	DMF	↓	↓	30	
01/29/07	DMF	↓	↓	30	
01/31/07	DMF	↓	↓	30	
02/06/07	DMF	↓	↓	30	
02/12/07	DMF	↓	↓	30	

Acceptance Range is 28-32 RPMS.
Meters Should Be Verified Against A Wrist Watch Annually And Recorded In The Comments Section.

QAQC276 0000003

TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

FIGURE 8

EXAMPLE PAGE FROM NON-VOLATILE TCLP EXTRACTION LOGBOOK

KATAHDIN ANALYTICAL SERVICES, INC.
Non-Volatile TCLP / SPLP Extraction Log

Katahdin Sample No.: SC0805-70 Extraction Type (Check one): [X] TCLP [ ] SPLP
Client: _____ Matrix: SL

I. SOLIDS DETERMINATION (Check one box below): **Performed by:** AJB **on** 2/23/05

[X] 100% wet solids - waste will obviously yield no liquid upon pressure filtration (proceed to Section II).
[ ] <100% solids (perform solids determination below):

A. Weight of filter	____ g	G. Weight of liquid phase (F-B)	____ g
B. Weight of filtrate vessel	____ g	H. Percent wet solids [(E-G)/E x 100]	____ %
C. Weight of weigh boat + waste	____ g	Note: Steps I - L are optional. Refer to SOP.	
D. Weight of weigh boat + residue	____ g	I. Weight of filter + dry solids	____ g
E. Total weight of waste (C-D)	____ g	J. Weight of dry solids (I - A)	____ g
F. Weight of filtrate vessel + filtrate	____ g	L. Percent dry solids (J/E x 100)	____ %

II. pH DETERMINATION AND FLUID SELECTION **Performed by:** AJB **on** 2/23/05

Initial pH of solid phase: 7.25 (For TCLP, if <5, proceed to Section III using TCLP Fluid #1. If >5, proceed to next step.)
TCLP only: pH after addition of 3.5 mL of 1 N HCl: 1.30 (If <5, use TCLP Fluid #1. If >5, use TCLP Fluid #2)
SPLP only: Sample from [ ] east (use SPLP Fluid #1) [ ] west (use SPLP Fluid #2) of Mississippi River.

III. EXTRACTION SETUP (Check one box below): **Performed by:** AJB **on** 2/23/05

[X] 100% wet solids: 2000 mL of Fluid # 1 (Batch 889) added to 100.02 g unfiltered waste.
[ ] <0.5% dry solids: _____mL of waste filtered (filter sufficient volume to support all required analyses).
[ ] >0.5% dry solids and <100% wet solids (perform phase separation below):
_____mL of Fluid # _____ (Batch _____) added to _____ g solid phase of waste.

M. Weight of filtrate vessel	____ g	Q. Weight of filtrate vessel + filtrate	____ g
N. Weight of weigh boat + waste	____ g	R. Weight of liquid phase (Q - M)	____ g
O. Weight of weigh boat + residue	____ g	S. Volume of liquid phase	____ mL
P. Total weight of waste (N - O)	____ g	T. Weight of wet solids (P - R)	____ g

Balance ID: Ohaus Galaxy 400
Associated Extraction Blank ID: PBT884L Fluid pH on day of use: 4.93
Extraction Bottle ID: #21 Fluid expiration date: 2/19/10

IV. ROTARY EXTRACTION CONDITIONS (Rotary extraction not required if waste <0.5% dry solids)

Rotary extractor ID: #3
Rotary extraction started: Date 2/23/05 Time 1515 Analyst AJB Room Temp. (degrees C) 19.7
Rotary extraction completed: Date_____ Time_____ Analyst_____ Room Temp. (degrees C)_____
Elapsed extraction time (HH:MM): _____:_____ pH of extract after extraction: _____
Rotary extraction filtered: Date_____ Analyst_____ Filter Lot Number_____
Was pre-extn. filtrate from Section III combined with rotary extract (check one)? [ ] Yes [ ] No [ ] N.A.
If no, enter the volume of filtrate obtained from the rotary extraction: _____mL
This TCLP/SPLP extract to be analyzed for (check all that apply):
[X] Metals [X] Semivolatiles [ ] Pesticides [ ] Herbicides [ ] Cyanide
Comments:

QAAA119 000003(

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS

Prepared By: Mike Thomas Date: 09/96

Approved By:

Group Supervisor: Michael F. Thomas Date: 11/15/00

Operations Manager: JC Bruta Date: 10/25/00

QA Officer: Deborah J. Nadeau Date: 10.24.00

General Manager: Dennis P. Hogan Date: 11/16/00

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Minor changes throughout. Clarifications to procedure section.	DN	10.24.00	10/24/00
02	Addition of compounds to Figure 2.	DN	3.28.02	3.28.02
03	Definitions added to section 1.1. Wording was added or changed to clarify sections 4, 5, 6, 7, 8 + 9. Minor changes throughout. New figures.	MRC	11.08.04	11.08.04
04	Updated Sect. 5.0 with current spike solutions prep. Removed section on medium level soil extraction. Replaced figure 3 and 4 with current LCS/MS spike components. Minor corrections to sect. 1.3, 4.24, 6.0 and 7.12. Updated logbook	LAD	04/06	04/06
05	Many changes made throughout, including but not limited to, waste information, updated spikes and surrogates, added SIM LCS/D and MS/D information, updated Table 1. Please refer to the QAM/SOP change form filed w/ SOP in QA for a detailed list of changes.	LAD	09/07	09/07

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Updated logbook example. Added adipate compounds to fig. 2. Added necessity of recording lot numbers of consumables in logbook. Added to record the temperature of the nitrogen evaporation water bath.	LAD	07/08	07/08
07	Added requirement to add spike before Na_2SO_4. Changed N_2 water bath temperature from < 39°C to < 30°C. Removed respirator reference. Added KAEHS manual. Added KAS SOP CA-1008 reference for additional subsampling information.	LAD	02/09	02/09

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-512-07**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-512-07**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS.**

Recipient: _____ Date: _____

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures and requirements for the preparation of solid samples for analysis of extractable semivolatile organic compounds. This SOP is specifically applicable to EPA Method 3550B in accordance with SW-846 Method 8270, current revision.

1.1 Definitions

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the extraction of samples for semivolatile analysis. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training”.

It is the responsibility of all Katahdin technical personnel involved in the extraction of samples for semivolatile analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab

notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to indicate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Waste Disposal

Wastes generated during the preparation of samples must be disposed of in accordance with the procedures described in the current revision of the Katahdin Analytical Environmental Health and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the rinsing of glassware etc. should be disposed of in the “D” waste stream satellite accumulation area nearest the point of generation. Acetone and methanol are considered flammable waste, and should be disposed of in the “O” waste stream satellite accumulation area nearest the point of generation. Post-extraction soil samples and sodium sulfate waste should be disposed of in the soil with organics “I” waste stream satellite accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

2.0 SUMMARY OF METHOD

A 30 gram portion of sediment/soil is mixed with anhydrous powdered sodium sulfate and extracted with 1:1 methylene chloride/acetone (v/v) using an ultrasonic probe. The methylene chloride extract is dried and concentrated to a volume of 1.0 mL.

3.0 INTERFERENCES

Contaminants in solvents, reagents, glassware, and other sample processing hardware may cause method interferences such as discrete artifacts and/or elevated baselines in the total ion current profiles (TICPs). All of these materials routinely must be demonstrated to be free from interferences under the conditions of the analysis by running laboratory method blanks. Interferences caused by phthalate esters can pose a major problem in semivolatile organics analysis because many phthalates are also target analytes. Common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory.

At no time may gloves that have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Additionally, whenever possible plastic items in this lab must be replaced with metal or Teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure clean glassware and apparatus are used, pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis. Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to inteferences, further cleanup of the sample extract may be needed to minimize interferences.

4.0 APPARATUS AND MATERIALS

Prior to use, all glassware must be rinsed three times with methylene chloride. Brand names and catalog numbers are included below for illustration purposes only.

4.1 Syringe - gas tight, 1.0 mL, solvent rinsed between each use.

4.2 Sonicator ultrasonic processor XL – Misonix (or equivalent) equipped with dual titanium 3/4" horn extenders for extracting two samples at a time.

4.3 Powder funnels, 100 mm diameter, 35 mm stem

4.4 Kuderna-Danish (KD) apparatus - Concentrator tube - 10 mL
Evaporative flask - 500 mL
Snyder column - 3-ball macro

4.5 Filter paper, 7.0 cm, Whatman #4

4.6 Vacuum filtration flask - 500 mL Erlenmeyer

4.7 Buchner funnel, porcelain, Coors® with 85 mm plate diameter (or equivalent)

4.8 Beakers - 400 mL

4.9 Boiling chips - approximately 12 mesh, silicon carbide (carborundum or equivalent). Soxhlet extract overnight in methylene chloride.

4.10 Water bath - eight position concentric ring bath, or equivalent, equipped with a calibrated thermometer. The bath should be used in a hood.

4.11 Balance - capable of accurately weighing ± 0.1 g.

4.12 Vials and caps – 1.8 mL with PTFE/silicone septa and 12 mL with Teflon-lined caps for extracts designated for GPC cleanup.

4.13 Filter paper, 18.5 cm, Fisherbrand or Whatman 114V (or equivalent)

4.14 Pasteur pipets - disposable, 5 ¾ ".

4.15 Nitrogen evaporation apparatus.

4.16 Muffle oven – capable of maintaining 400 °C for baking glass wool and organic-free sand.

5.0 REAGENTS

5.1 Sodium Sulfate - anhydrous powdered and granular crystals, reagent grade, certified by the manufacturer/vendor as purified heating to 400°C prior to receipt by the laboratory. Solvent rinse immediately prior to use by rinsing three times with pesticide grade methylene chloride. (Jost Chemical anhydrous powder, catalog #2797 or equivalent, and Jost Chemical granular crystals, catalog #2796 or equivalent).

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

5.2 Methylene chloride, methanol, and acetone - pesticide residue analysis grade or equivalent. Methylene chloride and acetone are evaluated by lot prior to use by concentration of approximately 400 mL to 1.0 mL followed by GC/MS analysis. The lot numbers of all solvents used during an extraction must be recorded in the extraction logbook.

5.3 Organic-free sand, purified by baking at 400 °C. Method blanks serve as checks on the baked sand.

5.4 Base/Neutral and Acid (SVOA) Surrogate Spiking Solution - Surrogate standards are added to all samples and calibration solutions. Prepare a surrogate standard spiking solution that contains the following compounds at the indicated concentrations in acetone.

Compound	Conc.
phenol-$_{d6}$	100 ug/mL
2,4,6-tribromophenol	100 ug/mL
2-fluorophenol	100 ug/mL
nitrobenzene-$_{d5}$	50 ug/mL
p-terphenyl-$_{d14}$	50 ug/mL
2-fluorobiphenyl	50 ug/mL

Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem. If reanalysis of a method blank still indicates surrogates out of criteria, a new surrogate solution must be used.

5.5 SIM Surrogate Spiking Solution- Surrogate Standards are added to all samples and calibration solutions. Prepare a surrogate solution that contains the following compounds at a concentration of 2 ug/mL in acetone.

Compound	Conc. ug/mL
Fluorene-d10	2.0 ug/mL
2-Methylnaphthalene-d10	2.0 ug/mL
Pyrene-d10.	2.0 ug/mL
2,4-Dibromophenol	2.0 ug/mL

Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem. If reanalysis of a method blank still indicates surrogates out of criteria, a new surrogate solution must be used.

5.6 Base/Neutral and Acid (SVOA) Matrix Spike/Lab Control Sample Spiking Solution - Prepare a spiking solution in methanol that contains the compounds listed in Figure 2 at a concentration of 50 ug/mL for base/neutrals and 100 ug/mL for acids. Store the

spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

5.7 Base/Neutral and Acid (SVOA APPENDIX IX) Matrix Spike/Lab Control Sample Spiking Solution. Prepare a spiking solution in methanol that contains the compounds listed in Figure 3 at a concentration of 100 µg/mL for each compound. Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem

5.8 Base/Neutral (SIM) Matrix Spike/ Lab Control Sample Spike Solution for SIM-SVOA. Prepare a spiking solution in methanol that contains the compounds listed in Figure 2 at a concentration of 2 ug/mL for base/neutral. Take out 1.0 mL of Base/Neutral and Acid Matrix Spike/Lab Control Spiking Solution for SVOA and dilute it to 25.0 mL in methanol. Store the solution Spiking at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Sediment/soil samples must be collected in a soil jar and must be maintained at 4°C (±2°C).

Holding time for extraction of sediment/soil samples for Method 3550 is 14 days from date of sample collection, although the analyst should be aware that actual holding times employed may be project/program specific.

Store all extracts at 4°C (±2°C) in the dark in labeled Teflon-sealed containers. See SOP SD-902, "Sample Receipt and Internal Control," current revision, for storage areas and temperature maintenance procedures.

7.0 PROCEDURES

Some solid samples may need to be cleaned up to reduce matrix interferences. The cleanup procedure employed is gel permeation chromatography (GPC). The organic department manager should be consulted to determine if a particular sample should be subjected to further cleanup procedures; the decision should consider sample history, sample appearance, and project/client needs.

Sign chain-of-custody when removing and replacing samples in storage locations, and fill out the sample preparation/extraction log with the necessary information before starting the extraction. Prerinse all glassware three times with methylene chloride.

7.1 Decant and discard any water layer on a sediment sample. Mix with a stainless steel spatula to ensure homogeneity of the sample. If the sample container is full to the extent that stirring the sample is impractical, try to remove the "best representative" aliquot from the jar based on color, particle size, moisture, etc. Remove any foreign objects such as sticks, leaves, and rocks, and note actions taken in the appropriate extraction logbook. Please refer to the current revision of Katahdin Analytical Services SOP CA-108, "Basic Laboratory Technique ", for more detailed guidance on subsampling to ensure reproducibility.

7.2 The following steps should be performed rapidly to avoid loss of the more volatile extractable. Weigh out a 30.0 ± 0.05 g portion of sample into a labeled 400-mL beaker. Record sample weight to the nearest 0.1 g in appropriate extraction logbook. Refer to Add between 30 g and 60 g of anhydrous powdered sodium sulfate as required for producing a "free-flowing" mixture. The amount of sodium sulfate added will depend upon the moisture content of the sample (e.g., low moisture content will require less sodium sulfate). Mix well with a spatula. Keep the spatula in the sample beaker and cover the beaker with aluminum foil.

7.3 A method blank must be prepared with each extraction batch, not to exceed 20 client samples. To prepare a method blank, weigh out one 30.0 ± 0.05 g portion of purified sand in a labeled 400 mL beaker. Refer to sections 7.7 and 7.7 for spike and surrogate addition instructions. Add 60 g sodium sulfate and mix well. Although a "free-flowing" mixture can be achieved with less than 60 g sodium sulfate, the method blank must contain 60 g in order to evaluate the sodium sulfate as a potential source of contamination.

7.4 A laboratory control sample (LCS) must be prepared with each extraction batch, not to exceed 20 client samples. To prepare LCS, weigh out one 30.0 ± 0.05 g portion of purified sand in a labeled 400 mL beaker. Refer to sections 7.7 and 7.7 for spike and surrogate addition instructions. Add 30 g sodium sulfate and mix well. If combined SIM-SVOA analysis is requested, a separate LCS must be prepared for each analysis. If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or Department Manager.

7.5 A matrix spike/matrix spike duplicate (MS/MSD) set must be prepared for every 20 samples. To prepare MS/MSD, weigh out 30.0 ± 0.05 g portions of the sample designated for MS/MSD into each of two labeled 400 mL beakers. Record sample weights to nearest 0.1 g in appropriate extraction logbook. Refer to sections 7.7 and 7.7 for spike and surrogate addition instructions. Add 30 - 60 g sodium sulfate to each to produce a free-flowing mixture, and mix well. If combined SIM-SVOA analysis is requested, a separate MS/MSD must be prepared for each analysis.

7.6 Record all weights to one decimal place in the extraction logbook.

7.7 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL of the appropriate base/neutral and acid surrogate spiking solution listed below using the pre-rinsed 1.0 mL gas tight syringe. The surrogate spike should be added **prior** to the addition of the sodium sulfate. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent prior to using it for another spiking solution.

7.7.1 If the request is for SVOA or SVOA Appendix IX, use the SVOA surrogate solution (sect. 5.4).

7.7.2 If the request is for SIM, use the SIM surrogate solution (sect. 5.5).

7.7.3 If the request is for SIM-SVOA, use both the SIM and SVOA surrogate solutions. NOTE: Separate LCS/LCSD and/or MS/MSD are needed for each analysis and should be spiked with the appropriate surrogate.

7.8 To the LCS/LCSD and the MS/MSD add 1.0 mL of the appropriate base/neutral and acid (SVOA) matrix spike/LCS spiking solution listed below using a 1.0 mL gas tight syringe. The LCS/MS spike should be added **prior** to the addition of the sodium sulfate. Record the matrix spike/LCS spiking solution volume and identification code in extraction logbook. Thoroughly rinse the syringe with solvent when spiking is completed.

7.8.1 If the request is for SVOA, add 1 mL of SVOA Spiking Solution (sect 5.6).

7.8.2 If the request is for SIM, add 1 mL SIM Spiking solution (sect 5.8).

7.8.3 If the request is for SVOA and SIM, add 1mL of SVOA Spiking Solution and 1 mL SIM Spiking solution (sect 5.6 and 5.8).

7.8.4 If the request is for SVOA Appendix IX, add 1mL of SVOA Spiking Solution and 1 mL of SVOA Appendix IX Spiking solution (sect 5.6 and 5.7).

7.9 To assure optimum operation and maximum energy output, the sonicators must be tuned daily prior to extracting samples. The following tuning procedure must be performed with the sonicator probes vibrating in air.

7.9.1 Turn OUTPUT CONTROL knob counter-clockwise to zero. This automatically switches the duty cycle to continuous mode.

7.9.2 Press and hold down the power switch to on.

7.9.3 Press and hold down the TUNE switch. Check if the counter is less or equal to 20%; otherwise, rotate the Tuning Knob (tuning button) clockwise until a reading of 20%or less is obtained.

7.9.4 Release the TUNE switch.

7.9.5 Turn OUTPUT CONTROL KNOB counter-clockwise to 50 and the power switch off.

7.9.6 Confirm that the sonicators were tuned by recording the date and/or percent in the extractions logbook.

7.10 Prior to extracting any samples, ensure that the sonicator probes are decontaminated by rinsing three times with a methylene chloride wash bottle. Collect the waste in a waste beaker. It may sometimes be necessary to wipe the upper part of each probe with a methylene chloride dampened KimWipe. Repeat this decontamination step between each sample on each probe.

7.11 To the mixed and spiked blank and LCS, add 100 mL of the 1:1 methylene chloride/acetone (V/V) solution and proceed with steps 7.11 through 7.14. Record the lot numbers of the solvents in the extraction logbook.

7.12 It may be necessary at this time to stir the sample/sodium sulfate mixture with the spatula to loosen up the mixture prior to extracting. Rinse the spatula with methylene chloride and collect the rinsing into a correspondent beaker. Position the beaker in the ultrasonic cell disruptor so that the bottom surface of the tip of the 3/4 inch disruptor horn is about halfway below the surface of the solvent and above the sediment layer.

7.13 Sonicate for 3 minutes with the output control knob set at 10, and mode switch on “pulsed” and % duty cycle knob set at 50%. While the mixture is sonicating, one should be able to see all, or most of the material, moving in the beaker under the influence of the energized probes. If not, stir the mixture again.

7.14 Prepare a filter flask fitted with a Buchner funnel. The Buchner funnel should contain a 7.0 cm Whatman #4 filter. Prerinse the flask, funnel and filter with methylene chloride and discard rinsings into solvent waste container. Decant extract into the filter flask and Buchner funnel. A vacuum pump may be used to facilitate filtration or the extract may be gravity filtered. The lot number of the filter paper must be written ti the extraction logbook.

7.15 Repeat the extraction two more times (sec 7.11 – 7.14) using 100 mL portions of 1:1 methylene chloride: acetone. Before each extraction, make certain that the sodium sulfate is still free-flowing and not a consolidated mass. As required, break up large

lumps with the spatula. Decant the extraction solvent into the Buchner funnel after each sonication. On the final sonication, pour the entire sample contents into the Buchner funnel and rinse thoroughly with methylene chloride to complete the quantitative transfer of the extract. Use the vacuum pump to pull all the extract into the flask

CONCENTRATION OF LOW LEVEL EXTRACTS

7.16 Rinse the K-D glassware (flask, concentration tube, and Snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride before assembling. Add two boiling chips to the K-D. Insert fluted 18.5 cm filter papers into short stem powder funnels and add ~ 2 inches of sodium sulfate crystals. Rinse the sodium sulfate in the assembled funnels with ~20-30 mLs of methylene chloride. Place the assembled K-D's under the funnels. The lot number of the filter paper must be written ti the extraction logbook.

7.17 Transfer the extracts to the K-D concentrator setups through the sodium sulfate in the funnels. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to pouring through the sodium sulfate. After pouring all of the extract volume through the sodium sulfate, rinse the extract flask three times with ~ 2 – 3 mls of methylene chloride. Add the rinsings through the sodium sulfate to complete a quantitative transfer. Rinse the sodium sulfate with ~ 15 mls of methylene chloride and allow to drain.

7.18 If samples are to be GPC'd, refer to the current revision of Katahdin SOP CA-513, Extract Cleanup Using Gel Permeation Chromatography, for appropriate concentrating procedures.

7.19 If samples are not to be GPC'd, follow Steps 7.19 through 7.24 to concentrate extracts to final volume of 1 mL. Otherwise proceed to GPC cleanup procedure as described in the current revision of Katahdin SOP CA-513.

7.20 Transfer the labels from the extract flasks to the K-Ds. Remove the funnel and attach a 3-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride.

7.21 Place the K-D in a hot water bath (75-85°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 6 mL, remove the K-D from the water bath. **Do not allow the evaporator to go dry. If the sample extract does go dry, re-extraction must occur immediately.** Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of methylene chloride. Remove the

Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL methylene chloride.

7.22 Reduce the extract in the concentrator tube to approximately 1 mL using the nitrogen blow-down apparatus. The bath temperature must be < 30°C. Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with ≈1 mL of methylene chloride. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N_2 sparging pipet closer to the surface of the extract to expedite the concentration. Record the temperature of the water in the nitrogen evaporation water bath in the logbook also note any problems or extract losses, if they occur, in the extractions logbook.

7.23 When the apparent volume reaches slightly less than 1 mL, remove the concentrator tube and allow it to cool.

7.24 Complete the quantitative transfer of the extract to a 1.8 mL vial by using methylene chloride. Adjust the volume of the methylene chloride extract to 1.0 mL using the 1.8 mL reference vial for volume comparison.

7.25 Label the vial with lab sample number, extraction date, matrix and analysis. Store extract vials at a temperature of 4 ± 2 °C until ready for analysis. Indicate in the extraction logbook the box number and “tray location” of the individual extract vials.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

A method blank must be extracted for each and every item listed below:

- Each sample matrix
- Each extraction method or level
- Every extraction batch of twenty or fewer samples

A laboratory control sample (LCS) is required for each and every item listed below:

- Each sample matrix
- Each extraction method or level
- Every extraction batch of twenty or fewer samples

Refer to the current revision of the applicable Katahdin SOP for analysis of Semivolatile Organics for quality control acceptance criteria.

Each extractions analyst must demonstrate proficiency in performing the extractions that prepare samples for analysis. Demonstration consists of preparation (extraction), by the analyst, of at least four aliquots which are then analyzed according to the analytical method in question. These QC samples must meet all quality control acceptance limits. Demonstration must be documented by use of a form which summarizes the results of the analysis of these aliquots, calculated percent recoveries, and standard deviation. Demonstration of proficiency must be done one time per analyst initially and then annually thereafter. Refer to SOPs QA-805 and QA-807, current revision.

If, upon analysis of the extracted samples, it is discovered that quality control acceptance criteria have not been met, all associated samples must be evaluated against all the QC. In some cases data may be reported, perhaps with narration, while in other cases, other corrective action may be taken. The corrective actions may include re-extraction of the samples associated with the quality control sample that did not meet acceptance criteria, or may include making new reagents and standards if the standardization is suspect. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Organic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit and Instrument Detection Limit Studies, for procedures on determining the MDL.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

Refer to the applicable analytical SOP for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste-Physical/Chemical Methods, Method 3550C, USEPA SW-846, Third Edition, Update IV, February 2007.

LIST OF TABLES AND FIGURES

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

TABLE 1

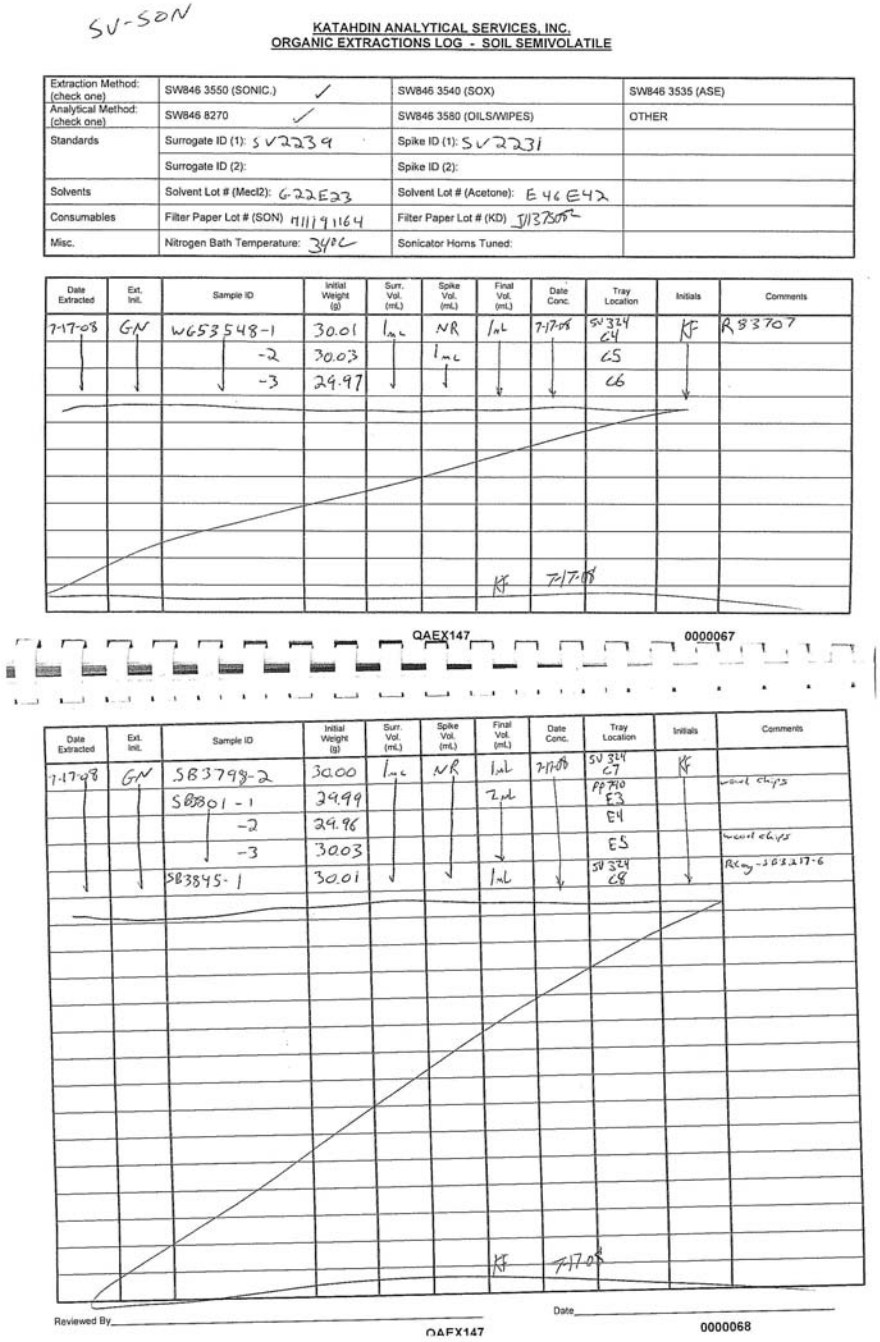
SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-512-07	METHOD 3550, current revision
Apparatus/Materials	1) short stem funnels	1) drying columns
Reagents		
Sample preservation/ handling		
Procedures	1) extract dried using Na_2SO_4 in short stem funnels 2) place sonicator horns ½ way between the surface of the solvent and the sediment layer 3) no apparatus height specification for concentration on water bath 4) water bath at 75-85 deg C 5) sample removed from water bath when volume reaches ~6 mL	1) extract dried using Na_2SO_4 in drying columns 2) place sonicator horns ½ inch below the solvent surface but above sediment layer 3) partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-15min 4) water bath at 80-90 deg C 5) sample removed from water bath when volume reaches 1-2 mL
QC - Spikes	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - LCS	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - Accuracy/Precision		
QC - MDL		

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

FIGURE 1

EXAMPLE OF LOGBOOK PAGE



SV-SON

KATAHDIN ANALYTICAL SERVICES, INC.
ORGANIC EXTRACTIONS LOG - SOIL SEMIVOLATILE

Extraction Method: (check one)	SW846 3550 (SONIC.) ✓	SW846 3540 (SOX)	SW846 3535 (ASE)
Analytical Method: (check one)	SW846 8270 ✓	SW846 3580 (OILS/WIPES)	OTHER
Standards	Surrogate ID (1): SV2239	Spike ID (1): SV2231	
	Surrogate ID (2):	Spike ID (2):	
Solvents	Solvent Lot # (MeCl2): G22E23	Solvent Lot # (Acetone): E46E42	
Consumables	Filter Paper Lot # (SON) 71119164	Filter Paper Lot # (KD) 7113805	
Misc.	Nitrogen Bath Temperature: 34°C	Sonicator Horns Tuned:	

Date Extracted	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spike Vol. (mL)	Final Vol. (mL)	Date Conc.	Tray Location	Initials	Comments
7-17-08	GN	W653548-1	30.01	1mL	NR	1mL	7-17-08	SV 324 C4	KF	R83707
		-2	30.03		1mL			C5		
		-3	29.97					C6		
						KF	7/17/08			

QAEX147 0000067

Date Extracted	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spike Vol. (mL)	Final Vol. (mL)	Date Conc.	Tray Location	Initials	Comments
7-17-08	GN	SB3798-2	30.00	1mL	NR	1mL	7-17-08	SV 324 C7	KF	
		SB3801-1	29.99			2mL		PP710 E3		Level chgs
		-2	29.96					E4		
		-3	30.03					E5		Level chgs
		SB3845-1	30.01			1mL		SV 324 C8		Reanj - SB5217-6
						KF	7/17/08			

Reviewed By_____ Date_____

QAEX147 0000068

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

FIGURE 2

LCS/MATRIX SPIKE COMPONENT LIST

BASE/NEUTRALS	
1-Methylnaphthalene	Bis (2-chloroethoxy) methane
1,1-Biphenyl	Bis (2-chloroethyl) ether
1,2,4-Trichlorobenzene	Bis (2-Chloroisopropyl) ether)
1,2-Dichlorobenzene	Bis (2-ethylhexyl) adipate
1,3-Dichlorobenzene	Bis (2-ethylhexyl) phthalate
1,4-Dichlorobenzene	Butylbenzyl phthalate
1,4-Dioxane	Caprolactam
2,4-Dinitrotoluene	Carbazole
2,6-Dinitrotoluene	Chrysene
2-Chloronaphthalene	Dibenz (a, h) anthracene
2-Methylnaphthalene	Dibenzofuran
2-Nitroaniline	Diethyl adipate
3,3'-Dichlorobenzidine	Diethyl phthalate
3-Nitroaniline	Dimethyl phthalate
4-Bromophenylphenyl ether	Di-n-butylphthalate
4-Chloroaniline	Di-n-octyl phthalate
4-Chlorophenylphenyl ether	Fluoranthene
4-Nitroaniline	Fluorene
Acenaphthene	Hexachlorobenzene
Acenaphthylene	Hexachlorobutadiene
Acetophenone	Hexachlorocyclopentadiene
Aniline	Hexachloroethane
Anthracene	Indeno (1,2,3-cd) pyrene
Atrazine	Isophorone
Azobenzene	Naphthalene
Benzaldehyde	Nitrobenzene
Benzidine	N-Nitrosodimethylamine
Benzo (a) Anthracene	N-Nitroso-di-n-propylamine
Benzo (a) pyrene	N-Nitrosodiphenylamine
Benzo (b) fluoranthene	Phenanthrene
Benzo (ghi) perylene	p-toluidine
Benzo (k) fluoranthene	Pyrene
Benzyl alcohol	Pyridine

ACIDS		
2, 3, 4, 6-Tetrachlorophenol	2-Chlorophenol	Benzoic acid
2,4,5-Trichlorophenol	2-Methylphenol	Ethyl methanesulfonate
2,4,6-Trichlorophenol	2-Nitrophenol	Methyl methanesulfonate
2,4-Dichlorophenol	4,6-Dinitro-2-methylphenol	Pentachlorophenol
2,4-Dimethylphenol	4-Chloro-3-methylphenol	Phenol
2,4-Dinitrophenol	4-Methylphenol	
2,6-Dichlorophenol	4-Nitrophenol	

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

FIGURE 3

APPENDIX IX LCS/MATRIX SPIKE COMPONENT LIST

1,2,4,5-Tetrachlorobenzene	Hexachloropropene
1,3,5-Trinitrobenzene	Isodrin
1,4-Naphthoquinone	Isosafrole
1-Chloronaphthalene	Kepone
1-Naphthylamine	m-Dinitrobenzene
2,4-D	Methapyrilene
2-Acetyl aminofluorene	Methyl parathion
2-Naphthylamine	n-Nitrosodiethylamine
2-Picoline	n-Nitrosodi-n-butylamine
3,3-Dimethylbenzidine	n-Nitrosomethylethylamine
3-Methylcholanthrene	n-Nitrosomorpholine
4-Aminobiphenyl	n-Nitrosopyrrolidine
4-Nitroquinoline-1-oxide	n-Nitrotrosopiperidine
5-Nitro-o-toluidine	O,O,O-Triethyl phosphorothioate
7,12-Dimethylbenz(a)anthracene	o-Toluidine
a,a-Dimethylphenethylamine	Parathion
Acetophenone	p-Dimethylaminoazobenzene
Aramite	Pentachlorobenzene
Chlorobenzilate	Pentachloronitriobenzene
Diallate	Phenacetin
Dibenz(a,j)acridine	Phorate
Dimethoate	p-Phenylenediamine
Dinoseb	Pronamide
Diphenylamine	Safrole
Disulfoton	Silvex (2,4,5-TP)
Famphur	Sulfotep
Hexachlorophene	Thionazin

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

Prepared By: Mike Thomas Date: 8/96

Approved By:

Group Supervisor: Michael F. Thomas Date: 11/15/00

Operations Manager: JCBurton Date: 10/25/00

QA Officer: Deborah J. Nadeau Date: 10·23·00

General Manager: Dennis F. Kuhn Date: 11/16/00

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Miner changes throughout. clarifications to procedure section.	DN	10·23·00	
02	Addition of SPE Procedure. minor changes throughout Added wording to sections 6 and 8	LAD	013105	013105
03	Added separate QC for Pest. and PCB. updated concentration procedure to reflect current practices. Changes in wording for clarification. Update Logbook page	LAD	04/06	04/06
04	Added waste generated and disposal info. Added missing definitions. Updated SPE extraction procedure. Updated Table 1 and 2. Added Table 3.	LAD	09/07	09/07
05	Updated logbook example. Added logbook requirements	LAD	09/08	09/08

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Added information for determining initial volume. Added reference to CA-108. Added clarification for LCS/D and MS/D sets for PEST/PCB analysis. Minor changes to reflect current techniques	LAD	10/09	10/09

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy _____ of document **SOP CA-515-06,** titled **PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy _____ of document **SOP CA-515-06,** titled **PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS**.

Recipient: _____ Date: _____

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures utilized by Katahdin Analytical Services, Inc. laboratory personnel for the preparation of aqueous samples prior to analysis for pesticides/PCBs by GC/ECD. It includes extraction of water samples by separatory funnel, continuous liquid-liquid, and solid phase extraction methods (EPA Methods 3510, 3520, 3535A, and EPA Method 608 current revisions).

1.1 Definitions

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of, analysts experienced in the extraction of aqueous samples for pesticides/PCBs analysis. Each analyst must demonstrate the ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training”.

It is the responsibility of all Katahdin personnel involved in the preparation of aqueous samples for pesticides/PCBs analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the

samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for the data.

It is the responsibility of the Supervisor to oversee that members of their group follow this SOP, that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their supervisor, or designee, appropriate for the job functions they will perform.

1.4 Waste Disposal

Wastes generated during the preparation of samples must be disposed of in accordance with the procedures described in the current revision of the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the rinsing of glassware etc. should be disposed of in the “D” waste stream satellite accumulation area nearest the point of generation. This includes the methylene chloride waste layer generated during CLLE extraction. Special care should be taken to pour this layer off into the appropriate waste stream, leaving the sample waste to be disposed of as follows. Since Pesticide/PCB samples are at a neutral pH, SEP funnel or CLLE sample waste may be dumped into either the “N-Hi” or “N-low” satellite accumulation area. Acetone and hexane are considered flammable waste, and should be disposed of in the “O” waste stream satellite accumulation area nearest the point of generation. Acid waste generated during the cleanup of PCB samples should be disposed of in the “O” satellite accumulation area nearest the point of generation. Please refer to

the current revision of SOP CA-107 for the location of satellite waste accumulation areas

2.0 SUMMARY OF METHOD

Pesticides/PCBs are extracted from aqueous samples using methylene chloride and separatory funnel, continuous liquid-liquid apparatus or Automated Extractor System (SPE), following EPA Methods 3510, 3520, 3535A and EPA Method 608. The methylene chloride is exchanged with hexane for the final extract. Method detection limit studies must be performed annually for pesticides/PCBs using all extraction methods, if the extraction lab wishes to use either or all techniques. Method 3510 (separatory funnel) is generally preferred for pesticides/PCBs since organochlorine pesticides may dechlorinate if under elevated pH conditions for an extended period of time. (Section 3.2, Method 3510B, Rev. 2, 9/94)

3.0 INTERFERENCES

Solvents, reagents, glassware, and other sample preparation apparatus may yield interferences to GC analysis due to the presence of contaminants. These contaminants can lead to discrete artifacts or elevated baselines in chromatograms. Routinely, all of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running reagent blanks. Interferences caused by phthalate esters can pose a major problem in pesticide analysis. Common flexible plastics contain varying amounts of phthalates which are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory. At no time may gloves which have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Additionally, whenever possible plastic items in this lab must be replaced with metal or Teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure clean glassware and apparatus are used, pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis.

Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to inteferences, further cleanup of the sample extract may be needed to minimize interferences.

4.0 APPARATUS AND MATERIALS

Prior to use, all glassware must be rinsed three times with the solvent to be used for extraction.

4.1 Separatory Funnel - 2000 mL capacity, Nalgene Teflon FEP separatory funnels with Nalgene Tefzel® screw-cap closures (or equivalent)

4.2 Concentrator tube - 10 mL, graduated

4.3 Evaporative flask - Kuderna-Danish, 500 mL capacity attached to concentrator with neck clips

4.4 Snyder column - Kuderna-Danish, three ball macro

4.5 Graduated cylinders - 100 mL, 1000 mL, or 2000 mL

4.6 Short Stem Funnels

4.7 250 mL amber collection bottles with Teflon-lined caps

4.8 12 mL and/or 16 mL glass vials with Teflon-lined caps

4.9 Continuous liquid-liquid extractors (CLLE) including body, 500 mL flat bottom boiling flask and Alhin condensers

4.10 Filter paper, 18.5 cm, Fisherbrand or Whatman 114V (or equivalent)

4.11 Nitrogen evaporation apparatus.

4.12 Boiling chips - approximately 10/40 mesh, Teflon or selenized carborundum, 12 mesh (or equivalent). Cleaned by Soxhlet.

4.13 Water bath - eight position concentric ring bath or equivalent, equipped with a calibrated thermometer.

4.14 Vials, 60 mL with PTFE – lined screw caps.

4.15 Horizon SPE-DEX 4790 Automated Extractor System.

4.16 Atlantic DVB disks, or equivalent.

4.17 1-L amber bottles

5.0 REAGENTS

5.1 Laboratory reagent grade water - water in which an interferent is not observed at or above the PQL for any parameter of interest (carbon filtered ASTM Type II water or equivalent)

5.2 Sodium Hydroxide (10N) – Purchased from vendor, “Baker-analyzed”, or equivalent

5.3 Sodium Sulfate (ACS) - Granular, anhydrous. Bake at 400°C for 4 hours (may be done by vendor). Purify by rinsing three times with pesticide grade methylene chloride. Allow residual methylene chloride to evaporate before use. Stored in a Teflon capped glass bottle.

5.4 Sulfuric Acid Solution (1:1) - Add 500 mL concentrated sulfuric acid (certified ASC grade or better) slowly to 500 mL laboratory reagent grade water. Prepare as needed and store in a ground glass stoppered bottle.

5.5 Methylene Chloride ($MeCL_2$) - Pesticide grade or better. Lot must be verified by concentrating 300-400 mL to 1.0 mL and evaluating by GC/MS.

5.6 Acetone and Hexane - Pesticide grade or better. Lot must be verified by concentrating approximately 20-30 mL to 1.0 mL and evaluating by GC/ECD.

5.7 Pesticide/PCB Surrogate spiking solution - Prepare a solution of decachlorbiphenyl (DCB) and tetrachloro-meta-xylene (TCMX) at a concentration of 1.0 ug/mL ea in acetone. Store the solution at –10 to -20 °C in a Teflon sealed container. Solution must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.

5.8 Pesticide Matrix Spike/Lab Control Sample spiking solution - Prepare a matrix spiking solution in pesticide grade methanol that contains all target analytes listed below:

ANALYTE	ug/mL
4,4’-DDT	0.5
4,4’-DDD	0.5
4,4’-DDE	0.5
Aldrin	0.5
Dieldrin	0.5
Endrin	0.5
Endrin Aldehyde	0.5
Endrin Ketone	0.5
Endosulfan I	0.5
Endosulfan II	0.5
Endosulfan Sulfate	0.5
alpha-BHC	0.5

ANALYTE (cont.)	ug/mL
beta-BHC	0.5
delta-BHC	0.5
gamma-BHC (Lindane)	0.5
Heptachlor	0.5
Heptachlor epoxide	0.5
Methoxychlor	0.5
alpha-Chlordane	0.5
gamma-Chlordane	0.5

5.9 PCB Matrix Spike/Lab Control Sample spiking solution - Prepare a matrix spiking solution in pesticide grade acetone that contains 5.0ug/ml ea of Aroclor® 1016/1260 mix (Restek catalog# 32039).

5.10 Store the spiking solutions at –10 to -20 °C in a Teflon sealed container. The solutions must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples are collected in 1 L amber bottles and held at 4 (±2) °C until time of extraction.

Holding time for extraction of aqueous samples for Methods 3510, 3520, and 3535 is 7 days from date of sample collection, although the analyst should be aware that actual holding times employed might be project/program specific.

7.0 PROCEDURES

The following information must be recorded in the extraction logbook.

- Extraction method
- Surrogate and spike IDs
- Lot numbers of all solvents, acids and bases, sodium sulfate, filter paper
- Nitrogen evaporation water bath temperature
- Sample pH if appicable
- Extraction and Concentration dates
- Extraction and Concentration analyst
- Sample ID or QC sample ID
- Initial and final volumes or weight
- Surrogate and spike amounts
- Any sample cleanup preformed
- Final extract tray location
- Any comments regarding the sample extraction (ie. Emulsion)

- Prep batch start time and end time
- CLLE start time and end time
- Lot number of the vials the concentrated extracts are stored in.

SEPARATORY FUNNEL SAMPLE EXTRACTION

If an emulsion prevents acceptable recovery or client history indicates samples may demonstrate matrix interence, then samples should be extracted by continuous liquid-liquid extraction (CLLE).

7.1 Rinse all glassware three times with methylene chloride prior to use.

7.2 Label a 2 L Teflon separatory funnel and a 250 mL amber collection bottle clearly. Label should include laboratory sample number, matrix, analyte, and extraction date. Be sure that the detachable stopcocks are secured to the separatory funnels before adding samples.

7.3 Measure the initial volume by comparing the meniscus of the sample with the reference bottle of the same bottle type. Please refer to SOP CA-108, "Basic Laboratory Technique", for the reference bottle verification procedure. Record the volume and any notable characteristics (e.g. color, presence of sediment, or odor) in the extraction logbook.

7.4 Transfer the contents of the sample bottle to a 2 L separatory funnel.

7.5 Transfer 1 L of laboratory reagent grade water to a 2 L separatory funnel. This serves as a method blank for the extraction batch. A method blank must be prepared for every daily extraction batch of twenty or fewer samples.

7.6 Transfer 1 L of laboratory reagent grade water to a 2 L separatory funnel for each analysis to be performed (pesticide and/or PCB). This will serve as a Laboratory Control Sample (LCS). When Pesticides and PCBs are extracted together, a LCS and LCSD set must be extracted for each analysis. An LCS is required for every daily extraction batch of twenty or fewer samples and each analysis. If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or Department Manager.

7.7 A matrix spike/matrix spike duplicate (MS/MSD) is to be prepared as requested by a client or, at a minimum, one pair per 20 samples or every 14 days and each analysis(refer to the logbook page, "date QC expires"). Transfer two additional 1 L aliquots of sample to 2 L separatory funnels for a matrix spike and matrix spike duplicate (MS/MSD) for each analysis. When Pesticides and PCBs are extracted together, a MS and MSD set must be extracted for each analysis. Note: Sufficient sample volume should be available without depleting all remaining sample aliquots.

7.8 Check the pH of the samples. If it is not between pH 5 and 9, adjust the pH with 10N sodium hydroxide or 1:1 sulfuric acid solution. Note the addition of NaOH or H_2SO_4 in the extraction logbook.

7.9 Using a gas-tight syringe, add 1.0 mL of surrogate spiking solution to all samples the blank, LCS/LCSD(s) and MS/MSD(s), if performed.

7.10 Using a gas-tight syringe, add 1.0 mL of pesticide or PCB matrix spiking solution to the appropriate LCS, LCSD, MS and MSD if performed.

7.11 To each empty sample bottle add 60 mLs of methylene chloride, rinse the bottle and transfer the solvent into the appropriate separatory funnel. Add 60 mL of methylene chloride directly to the blank and LCS/LCSD(s).

7.12 Ensure that each screw cap is secured tightly to the separatory funnel to prevent leaks. Shake briefly and vent in hood to release pressure. Extract the sample by shaking the funnel on mechanical shaker for 3 minutes. Allow phases to separate for at least 10 minutes. Drain the methylene chloride layer into the 250 mL amber collection bottle.

7.13 If an emulsion forms, mechanical techniques must be employed to achieve maximum separation and solvent recovery. Such means include swirling and centrifugation and draining through a small separatory funnel. In certain instances, transferring the entire sample into a continuous liquid-liquid extractor may be the only alternative. If any such techniques are used, they must be noted in the extractions logbook.

7.14 Add a second 60 mL aliquot of methylene chloride to the separatory funnel and extract for the second time (see 7.10 - 7.12). Collect the methylene chloride layer in the same 250 mL amber collection bottle.

7.15 Repeat the extraction for a third time as described in 7.13.

7.16 Proceed to Section 7.53 for extract concentration procedures.

CONTINUOUS LIQUID-LIQUID SAMPLE EXTRACTION (CLLE)

7.17 Set up the CLLE apparatus. All glassware should be rinsed three times with methylene chloride and the extract flasks properly labeled.

7.18 Add 2-3 boiling stones to the round bottom flask and approximately 500 - 600 mL of methylene chloride to the CLLE body.

7.19 Add 1 L laboratory reagent grade water to a CLLE body. This is the method blank for this extraction batch. Be sure that no water leaks into the round bottom flask. A method blank is required for every extraction batch of twenty or fewer samples.

7.20 Mark the sample level (meniscus) on the sample bottle with a wax crayon so that the volume can be measured (this may be done prior to removal from the walk-in cooler). Transfer the sample to a CLLE body, being sure that no water leaks into the round bottom flask.

7.21 Prepare an LCS for every daily extraction batch of twenty or fewer samples and each analysis (pesticide and/or PCB). Add 1 L of laboratory reagent grade water to a CLLE body. If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or Department Manager. When Pesticides and PCBs are extracted together, a LCS and LCSD set must be extracted for each analysis.

7.22 Mark the sample levels on the sample bottles. Transfer the samples to the CLLE bodies.

7.23 Check the pH of the samples. If it is not between pH 5 and 9, adjust the pH with 10N sodium hydroxide or 1:1 sulfuric acid solution. Note the addition of NaOH or H_2SO_4 in the extraction logbook.

7.24 Transfer two 1 L portions of a sample to CLLE bodies for each analysis for preparation of a matrix spike/matrix spike duplicate if required. An MS/MSD is required if requested by the client or per 20 samples, whichever occurs first. When Pesticides and PCBs are extracted together, a MS and MSD set must be extracted for each analysis. Note: Sufficient sample volume should be available without depleting all remaining sample aliquots.

7.25 For each sample, rinse the original sample container with approximately 30 mL of methylene chloride. Add this rinse to the CLLE body.

7.26 Determine the initial volume of the samples by comparing the grease marking where the sample meniscus was to the reference bottle located in the lab. Please refer to SOP CA-108, “Basic Laboratory Technique”, for the reference bottle verification procedure. Record the volume and any notable characteristics (e.g. color, presence of sediment, or odor) in the extraction logbook.

7.27 Add 1.0 mL of the Pesticide/PCB Surrogate Spike to each sample including the blank, LCS/LCSD and MS/MSD, if performed.

7.28 Add 1.0 mL of Pesticide or PCB Matrix Spike to the appropriate LCS/LCSD and MS/MSD pair, if performed, and stir.

7.29 Attach cooling water Allihn condensers, after first rinsing each 45/50 joint with methylene chloride. Turn on the heating mantles and allow the samples to extract for at least 18 hours, total extract time may go up to 20 hours. Turn off the mantles and let samples cool.

7.30 Proceed to Section 7. 53 for sample extract concentration procedures.

EXTRACTION WITH AUTOMATED EXTRACTOR SYSTEM (SPE)

Alternatively, samples may be extracted using the Horizon Automated Extractor System (Figure 2)

Purging the Extractor Vessels

7.31 Check and fill all necessary solvent bottles (acetone, laboratory reagent grade water and hexane) as needed. Check and empty the two waste containers as needed.

7.32 Turn on nitrogen tank to 60 psi. Turn the instrument pressure on top of the controller to 50 psi. Turn the solvent bottle pressure to 10 psi.

7.33 Turn on the Horizon controller (switch in the back).

7.34 Check the lubrication oil on the air pump. Fill as needed. Turn the air pump on.

7.35 Clean the glass sensors that are located on the back of the dispensing stems of the extractors using a Kim Wipe. This is to remove any residue that may interfere with the sensors.

7.36 Attach 19/22 adapters to 40-mL vials and attach beneath the disk holder platforms of the extractors. Assembly per owner's manual and place empty Horizon disk holder assemblies on top of the disk holder platforms. There should be roughly 1 cm separating the speedisk from the extractor downtube.

7.37 Check to be sure that all extractors have empty sample bottles loaded on top. If not, use a Horizon cap on a one liter empty bottle and firmly place the bottlenose down into the extractor.

7.38 Press *select* on the control panel to designate an extractor (1, 2, 3, 4 or "." for all), then press *enter*.

7.39 Type 8081.9, and press enter to select pesticide/PCB purge method. Once the method is loaded, start the extractors by pressing the *start* buttons on the individual extractors. The red LED will blink when the method is complete.

7.40 Repeat this process 2-3 times before using the Horizon autoextractors.

ANALYSIS OF SAMPLES WITH AUTOEXTRACTOR

7.41 Label a 60 ml vial with the sample to be extracted. Attach 19/22 adapter to vial and attach beneath the disk holder platforms of the extractors.

7.42 Place an Atlantic DVB disk (or equivalent) into a Horizon disk holder assembly and assemble per owner's manual. Place the disk holder assemble on top of the disk holder platform. There should be roughly 1 cm separating the speedisk from the extractor downtube

7.43 Mark the volume level of liquid in each sample on the outside using a grease pencil.

7.44 Add 1 L laboratory reagent grade water to 1 L amber bottle. This is the method blank for this batch. A method blank is required for every extraction batch of twenty or fewer samples.

7.45 Prepare an LCS for every daily extraction batch of twenty or fewer samples and each analysis, pesticide and/or PCB. Add 1 L of laboratory reagent grade water to a 1 L amber bottle. If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required to meet client specific or program specific requirements. This information will be disseminated from the project manager or department manager.

7.46 Add 1.0 mL of Pesticide/PCB surrogate spike to each sample including the blank, LCS/LCSD and MS/MSD, if required. Recap samples and shake well.

7.47 Add 1.0 mL of pesticide or PCB matrix spike to the appropriate LCS/LCSD and MS/MSD samples. Recap and shake well.

7.48 Remove cap and add 5.0 mL of 1:1 H_2SO_4 to each sample including the blank, LCS/LCSD and MS/MSD set immediately prior to extracting the sample.

7.49 Remove the cap from each sample bottle and cover with tin foil. Screw a Horizon adapter cap over the tin foil. Invert the bottle and check for leaks.

7.50 Load the sample bottle on the holder and twist ¾ of a rotation. Stop twisting when air bubbles rise to the top of the sample bottle. Do not twist completely around. The foil may loosen and jam the valve.

7.51 Press *select* on the control panel to designate an extractor (1, 2 or "." for both), then press *enter*.

7.52 Type in 8081.3 for the method and press enter. Once the method is loaded, start the extractors by pressing the *start* buttons on the individual extractors. The red LED will blink when the method is complete. The extract will be collected in the 60 ml vial.

7.53 Sample is now ready to reduce to 10 mL final volume.

NOTE: The instrument methods stated above apply specifically to the Atlantic DVB disk. Instrument methods may need to be modified with the usage of different filters and/or to increase recoveries. See instrument logbook for current methods in use.

CONCENTRATION OF WATER SAMPLE EXTRACTS

7.54 Rinse the K-D glassware (flask, concentration tube, funnel and Snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride (or hexane for samples extracted with the Autoextractor) before assembling. Add two boiling chips to the K-D. Insert fluted 18.5 cm filter papers into short stem powder funnels and add ~ 2 inches of sodium sulfate crystals. Rinse the sodium sulfate in the assembled funnels with ~20-30 mLs of methylene chloride (hexane for samples extracted with the Autoextractor). Place the assembled K-D's under the funnels.

7.55 For methylene chloride extracts, add approximately 50 mL Hexane to funnel and let drain through. Since methylene chloride has a lower boiling point than Hexane, this will result in a final extract in hexane only.

7.56 Transfer the methylene chloride or hexane extracts to the K-D concentrator setups through the sodium sulfate in the funnels. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to pouring through the sodium sulfate. After pouring all of the extract volume through the sodium sulfate, rinse the extract bottle three times with ~ 2 – 3 mLs of methylene chloride(or hexane for samples extracted with the Autoextractor). Add the rinsings through the sodium sulfate to complete a quantitative transfer. Rinse the sodium sulfate with ~ 15 mLs of methylene chloride (or hexane for samples extracted with the Autoextractor) and allow to drain.

7.57 Transfer the labels from the collection bottles or round bottom flasks (from the CLLE extraction) to the K-Ds. Remove the funnel and attach a 3-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride (or hexane for samples extracted with the Autoextractor).

7.58 Place the K-D in a hot water bath (85-90°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 5-6 mL, remove the K-D from the water bath. Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of hexane. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL hexane.

7.59 Reduce the extracts to ≈ 1 mL using Nitrogen blow-down apparatus. The bath temperature must be no higher than the boiling point of the solvent (45 °C for hexane). Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with ≈1 mL of methylene chloride. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N_2 sparging needle closer to the surface of the extract to expedite the concentration. Note any problems or extract losses, if they occur, in the extractions logbook. Transfer extract to a 12 or 16 mL vial. Using a reference vial for volume comparison, adjust the final extract volume to 10 mL by rinsing sides of tube with hexane and transferring rinsings to vial.

7.60 If at any point in the concentration procedure the concentrator tube goes dry – reextract the sample immediately.

7.61 Transfer the label from the concentrator tube to the vial. Store extract vials at a temperature of 4 ± 2 °C until ready for analysis. Indicate in the extractions logbook the box number and “tray location” of the individual extract vials.

7.62 All sample extracts for 8082 PCB analysis must undergo a sulfuric acid wash (cleanup) prior to analysis. All sample extracts for 8081 pesticide analysis do not undergo further cleanup unless requested by the client. Therefore, all sample extracts for combined 8081/8082 analyses must be split. Prior to splitting, mix contents of vial well. One portion must be acid cleaned for 8082 analysis. The associated method blank must be split and acid-cleaned in the same fashion. PCB LCSs and matrix spikes are acid cleaned also. Pesticide LCSs and matrix spikes are not subjected to further cleanup. Please refer to Katahdin SOP CA525 (current revision), Extract Cleanup Using Sulfuric Acid, for further instructions.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Each extractions analyst must demonstrate proficiency in performing the extractions that prepare samples for analysis. Demonstration consists of preparation (extraction), by the analyst, of at least four aliquots which are then analyzed according to the analytical method in question. These QC samples must meet all quality control acceptance limits. Demonstration must be documented by use of a form which summarizes the results of the analysis of these aliquots, calculated percent recoveries, and standard deviation. Demonstration of proficiency must be done one time per analyst initially and then annually thereafter. Refer to SOPs QA-805 and QA-807, current revision.

If, upon analysis of the extracted samples, it is discovered that quality control acceptance criteria have not been met, all associated samples must be evaluated against all the QC. In some cases data may be reported, perhaps with narration, while in other cases, other

corrective action may be taken. The corrective actions may include re-extraction of the samples associated with the quality control sample that did not meet acceptance criteria, or may include making new reagents and standards if the standardization is suspect. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

A method blank must be extracted for each and every item listed below:

- Each day of extraction (24 hours midnight - midnight)
- Each extraction method
- Every 20 samples extracted in a 24-hour period

A laboratory control sample (LCS) is required for each and every item listed below:

- Each extraction method
- Every extraction batch of twenty or fewer samples
- Each analysis (pesticide and/or PCB) to be performed

Refer to the current revision of the applicable Katahdin SOP for analysis of Pesticides and PCBs for quality control acceptance criteria.

9.0 METHOD PERFORMANCE

Refer to the applicable analytical SOP.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste - Physical/Chemical Methods, Methods 3510C and 3520C, USEPA SW-846, Third Edition, Final Update III, December 1996.

40 CFR 136, Appendix A, "Test Procedures for Analysis of Organic Pollutants," Method 608, June, 1998 edition.

LIST OF TABLES AND FIGURES

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

TABLE 1

SUMMARY OF METHOD MODIFICATIONS (METHOD 3510, current revision)

TOPIC	KATAHDIN SOP CA-515-06	METHOD 3510, current revision
Apparatus/Materials	1. 12 or 16 mL vials used for final extract 2. 250 mL amber bottle or flask used 3. 1.0 mL syringe 4. short stem funnels	1. 2 mL vials used for final extract 2. 250 mL Erlenmeyer flask 3. 5.0 mL syringe 4. drying column
Reagents		
Sample preservation/ handling	1. entire contents of 1 L sample bottle transferred to separatory funnel	1. one liter graduated cylinders used to transfer initial sample volume to separatory funnel
Procedures	1. extract collection in amber bottle or Erlenmeyer flask 2. extract dried using Na_2SO_4 in short stem funnels 3. no apparatus height specification for concentration on water bath 4. sample removed from water bath when volume reaches ~10 mL 5. hexane added directly to K-D body at start of concentration process	1. extract collection in Erlenmeyer flask 2. extract dried using Na_2SO_4 in drying columns 3. partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-15min 4. sample removed from water bath when volume reaches 1-2 mL 5. solvent exchange via large K-D with addition of 50 mL hexane after concentrating methylene chloride extract to 1 mL
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

TABLE 2

SUMMARY OF METHOD MODIFICATIONS (METHOD 3520, current revision)

TOPIC	KATAHDIN SOP CA-515-06	METHOD 3520, current revision
Apparatus/Materials	1. short-stem funnels 2. 12 or 16 mL vials used for final extract	1. drying columns 2. 2 mL vials used for final extract
Reagents		
Sample preservation/ handling	1. entire contents of 1 L sample bottle transferred to CLLE	1. one liter graduated cylinders used to tranfer initial sample volume to CLLE
Procedures	1. CLLE for 18 $\pm$ 2 hours 2. extract dried using Na_2SO_4 in short stem funnels 3. no apparatus height specification for concentration on water bath 4. sample removed from water bath when volume reaches ~10 mL 5. hexane added directly to K-D body at start of concentration process	1. CLLE for 18-24 hours 2. extract dried using Na_2SO_4 in drying columns 3. partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-15min 4. sample removed from water bath when volume reaches 1-2 mL 5. solvent exchange via macro K-D with addition of 50 mL hexane after concentrating methylene chloride extract to 1 mL
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

TABLE 3

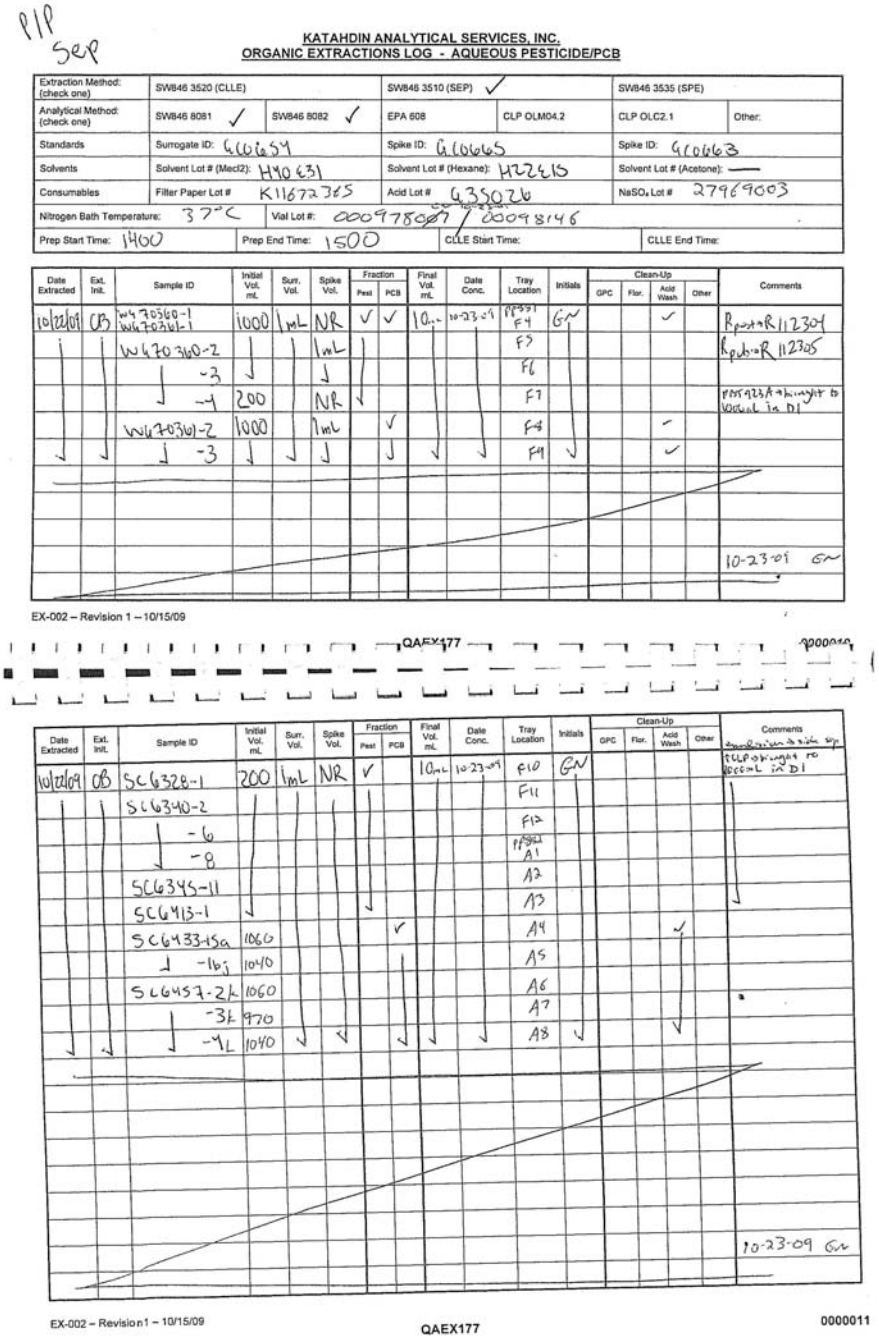
SUMMARY OF METHOD MODIFICATIONS (METHOD 3535, current revision)

TOPIC	KATAHDIN SOP CA-515-06	METHOD 3520, current revision
Apparatus/Materials	1. Horizon SPE-DEX 4790 Automated Extractor System.	1. Empore solid-phase extraction system
Reagents		
Sample preservation/ handling	1. entire contents of 1 L sample bottle transferred to separatory funnel	1. one liter graduated cylinders used to transfer initial sample volume to separatory funnel
Procedures	1. no methanol addition 2. extraction using Horizon SPE-DEX 4790 Automated Extractor System. 3. extract dried using Na_2SO_4 in short stem funnels 4. no apparatus height specification for concentration on water bath 5. sample removed from water bath when volume reaches ~10 mL 6. hexane added directly to K-D body at start of concentration process	1. 5mL methanol added to all samples and blanks 2. extraction using Empore solid-phase extraction system 3. extract dried using Na_2SO_4 in drying columns 4. partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-15min 5. sample removed from water bath when volume reaches 1mL 6. solvent exchange via macro K-D with addition of 50 mL hexane after concentrating methylene chloride extract to 1 mL
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

FIGURE 1

EXAMPLE OF LOGBOOK PAGE



PIP
Sep

KATAHDIN ANALYTICAL SERVICES, INC.
ORGANIC EXTRACTIONS LOG - AQUEOUS PESTICIDE/PCB

Extraction Method: (check one)	SW846 3520 (CLLE)		SW846 3510 (SEP) ✓		SW846 3535 (SPE)	
Analytical Method: (check one)	SW846 8081 ✓	SW846 8082 ✓	EPA 608	CLP OLM04.2	CLP OLC2.1	Other:
Standards	Surrogate ID: GC0654		Spike ID: GC0665		Spike ID: GC0663	
Solvents	Solvent Lot # (MeCl2): H40631		Solvent Lot # (Hexane): H22615		Solvent Lot # (Acetone): —	
Consumables	Filter Paper Lot # K11672365		Acid Lot # 435026		$NaSO_4$ Lot # 27969003	
Nitrogen Bath Temperature: 37°C		Vial Lot #: 00097808[illegible] / 00098146				
Prep Start Time: 1400	Prep End Time: 1500		CLLE Start Time:		CLLE End Time:	

Date Extracted	Ext. Init.	Sample ID	Initial Vol. mL	Surr. Vol.	Spike Vol.	Fraction: Pest	Fraction: PCB	Final Vol. mL	Date Conc.	Tray Location	Initials	Clean-Up: GPC	Clean-Up: Flor.	Clean-Up: Acid Wash	Clean-Up: Other	Comments
10/22/09	OB	W6703601 W6703611	1000	1mL	NR	✓	✓	10mL	10-23-09	PP991 F4	GN			✓		Repeat of R112304
↓	↓	W6703602	↓	↓	1mL	↓		↓	↓	F5						Repeat of R112305
		↓ -3	↓		↓					F6						
		↓ -4	200		NR	✓				F7						PP9923 A 4 brought to volume in DI
		W6703612	1000		1mL		✓			F8				–		
↓	↓	↓ -3	↓	↓	↓		↓	↓	↓	F9	↓			✓		
																10-23-09 GN

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0000010

Date Extracted	Ext. Init.	Sample ID	Initial Vol. mL	Surr. Vol.	Spike Vol.	Fraction: Pest	Fraction: PCB	Final Vol. mL	Date Conc.	Tray Location	Initials	Clean-Up: GPC	Clean-Up: Flor.	Clean-Up: Acid Wash	Clean-Up: Other	Comments
10/22/09	OB	SC6328-1	200	1mL	NR	✓		10mL	10-23-09	F10	GN					TCLP extract brought to volume in DI
		SC6340-2								F11						
		↓ -6								F12						
		↓ -8								PP992 A1						
		SC6345-11								A2						
		SC6413-1	↓			↓				A3						
		SC6433-15a	1060				✓			A4				✓		
		↓ -16j	1040				↓			A5						
		SC6457-2L	1060							A6						
		-3L	970							A7						
↓	↓	↓ -4L	1040	↓	↓		↓	↓	↓	A8	↓			↓		
																10-23-09 GN

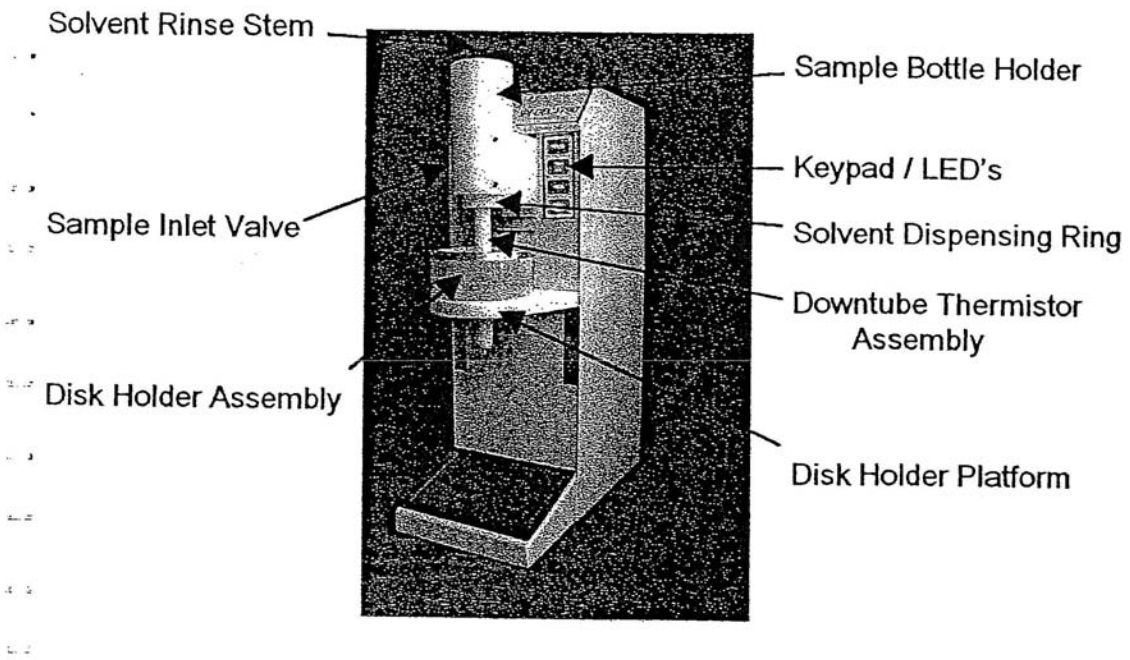
EX-002 – Revision 1 – 10/15/09

QAEX177

0000011

FIGURE 2

HORIZON AUTOEXTRACTOR SYSTEM DIAGRAM



KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-516
Revision History
Cover Page
Page 1

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR HERBICIDE ANALYSIS BY METHOD 8151

Prepared By: Keith Tanguay Date: 7/98

Approved By:

Group Supervisor: Michael F. Thomas Date: 1/26/01

Operations Manager: Joh C. Burton Date: 1/26/01

QA Officer: Deborah J. Nadeau Date: 1.26.01

General Manager: Dennis F. Hogan Date: 1/29/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 8151A	Format changes, added pollution prevention, other minor changes to sections 7+8.	DH	1.26.01	1/26/01
02 8151A	Wording was added or changed to clarify sections 6, 7, 8 & 9. Minor changes throughout.	MRC	11.08.04	11.08.04
03 8151A	Sect. 7.0 - added information regarding TCLP samples. Small changes to reflect current practices. Updated Logbook page.	LAD	04/06	04/06
04 8151A	Sect. 7.4 & 7.10: changed glassware rinse solvent from ether to MeCl2. Sect. 7.5: Sample volume determined by comparison to reference bottle & added 800ml of DI water is added to TCLP samples. Sect. 7.10 & 7.11: time shaken from 4 to 3 min. 7.17: removed old way of determining sample vol. 7.18: Added it may be necessary to add more NaOH. 7.22: changed Vol. Sample Kd's to. 7.31: Added additional info to be recorded in logbook. Typos and formatting fixed.	LAD	03/08	03/08
05	Sect. 5.2 - Added wording to clarify acidification procedure. Sect. 7.13 - Removed necessity to reduce acid amt. for TCLP samples. Sect. 7.18 - added fume hood. Sect. 7.32 added NaOH and NaCl2 lot #'s. Updated f.g. 1 - logbook page.	LAD	05/09	05/09

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR HERBICIDE ANALYSIS - METHOD 8151

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy _____ of document **SOP CA-516-05,** titled **PREPARATION OF AQUEOUS SAMPLES FOR HERBICIDE ANALYSIS BY METHOD 8151.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy _____ of document **SOP CA-516-05,** titled **PREPARATION OF AQUEOUS SAMPLES FOR HERBICIDE ANALYSIS BY METHOD 8151.**

Recipient: _____ Date: _____

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR HERBICIDE ANALYSIS - METHOD 8151

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures, based on EPA SW 846 method 8151, used by Katahdin Analytical Services, Inc. technical personnel for the extraction of chlorinated phenoxy acid herbicides from aqueous samples such as surface, well and discharge waters. Detection limits are at the ug/L level or greater

1.1 Definitions

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

DCAA: Dichlorophenylacetic acid

2,4-D: 2,4-Dichlorophenoxy acetic acid

ETHER: Diethyl ether- unpreserved

2,4,5-TP (Silvex): 2,4,5-Trichlorophenoxypropionic acid

2,4,5-T : 2,4,5-Trichlorophenoxyacetic acid

DIAZALD (a.k.a. Diazogen®): 99% (N-methyl-N-nitroso-p-toluenesulfonamide) See cautions in 1.3 Safety

CARBITOL: 2-(2-Ethoxyethoxy)ethanol

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the extraction of herbicides from aqueous samples. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

It is the responsibility of all Katahdin technical personnel involved in the extraction of herbicides from aqueous samples to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

This procedure requires the use of materials that, if handled improperly, pose a potential health risk to everyone in the laboratory. Follow instructions that describe the use of commercially available peroxide test strips for Diethylether. Special care must be taken when working with diazomethane.

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure. (See cautions prior to 7.24.)

Each qualified analyst or technician must be familiar with Katahdin Analytical Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of a respirator and all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Plan for further details on pollution prevention techniques.

Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Hazardous Waste Plan and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.
To minimize ether exposure in the laboratory, allow glassware to air dry in a fume hood before bringing to dish washing area

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR HERBICIDE ANALYSIS - METHOD 8151

2.0 SUMMARY OF METHOD

Chlorinated phenoxy acids and their esters are initially exposed to an alkaline hydrolysis at pH>12 and serially extracted three times with methylene chloride to remove chlorinated hydrocarbons and phthalate esters. The hydrolyzed sample then undergoes a pH adjustment to pH<2 and is extracted with diethyl ether. The diethyl ether extract is collected in a 500 mL screw cap bottle that contains approximately 20 grams of acidified anhydrous sodium sulfate. After drying for a minimum of two hours, the extract is concentrated to 1 ml. The 1 ml extract is brought up to 4 mls with the addition of 1 ml of isooctane, 0.5 ml of methanol and 1.5 mls of diethyl ether. The 4 ml extract then undergoes diazomethane esterification (methylation) and is subsequently analyzed by GC-ECD. Compounds of interest are detected as methyl esters.

3.0 INTERFERENCES

Organic acids, especially chlorinated acids, cause the most direct interference. Phenols, including chlorophenols, also may interfere. Alkaline hydrolysis and subsequent extraction eliminate many of the predominant chlorinated insecticides. Because the herbicides react readily with alkaline substances, loss may occur if there is alkaline contact at any time except in the controlled alkaline hydrolysis step. Glassware and glass wool should be acid-rinsed and sodium sulfate (Na_2SO_4) should be acidified to minimize any alkaline contact.

4.0 APPARATUS AND MATERIALS

4.1 2 L Separatory Funnel, Teflon FEP with screw closures

4.2 Glass rod for crushing Na_2SO_4

4.3 pH paper (0-14)

4.4 gas tight volumetric syringes, 1.0 mL, 0.5 mL

4.5 mechanical separatory funnel shaker

4.6 Water/Steam bath (for K-D solvent evaporation) Organomation S-Evap Model 120

4.7 Kuderna-Danish apparatus:

- Concentrator tube (or collector), 10 mL graduated
- Evaporator flask, 500 mL
- Three ball macro Snyder column

4.8 Nitrogen blow-down apparatus (for concentrating extracts in 10 mL concentrator tubes)

4.9 Pasteur pipets, Pasteur pipettes, 5 ¾"

4.10 12 mL vials

4.11 500 mL sample bottles

4.12 Scoopula(s)

4.13 Graduated cylinders, 25 mL, 100 mL, 1000 mL

4.14 Diazomethane Generator (See figure 2)

4.15 Boiling chips, teflon, or silicon carbide, (carborundum, 2 mesh)

4.16 Clean sodium sulfate jars (~2Liters) for collecting the aqueous phase.

5.0 REAGENTS

5.1 Potassium hydroxide (37%): Prepare by dissolving 37 g of potassium hydroxide pellets in DI water and diluting to 100 mL.

5.2 Acidified Anhydrous Sodium Sulfate: Prepare by adding hexane to a 2.5 Kg jar of sodium sulfate crystals until the crystals are completely submerged. Measure 25mL of concentrated hydrochloric acid or sulfuric acid with a graduated cylinder and add it to the hexane saturated salt crystals. Quickly stir the mixture with a glass rod until the sodium sulfate is loose. Then decant the solvent layer and transfer the sodium sulfate on to a sheet of aluminum foil under a hood. Let dry overnight and then transfer back to the original jar. Label jar as acidified sodium sulfate. Record date and initials on jar. Cover and store at room temperature. Acidified anhydrous sodium sulfate will be referred to as sodium sulfate further in this SOP.

5.3 Sulfuric acid (1+3): Prepare by slowly adding 25 mL of sulfuric acid to 75 mL of DI water. This dilution should be done within an ice water jacket; store the diluted acid in a laboratory refrigerator.

5.4 Herbicide surrogate solution containing 5 □g/mL DCAA acid in acetone.

5.5 Underivitized Chlorinated Herbicide Stock Solutions: Contains 18 compounds at various concentrations - 100 ug/ml for all except for MCPA and MCPP which are at 10,000 ug/ml. Dilute to 5.0 ug/ml and 500 ug/ml with acetone.

5.6 Diazald solution: weigh out ~6.8 grams of Diazald into a 50 mL volumetric flask and dissolve in a mixture of 25 mL carbitol and 25 mL of ether (50 mL of 1:1 v/v carbitol/ether). Bring to the volume mark only after all of the Diazald is in solution. Use the sonicator bath briefly if necessary. This solution is stable if kept at -10 - 20°C for one month.

5.7 Acetone: pesticides residue grade or equivalent.

5.8 Ether: pesticide residue grade or equivalent (unpreserved).

5.9 Methanol: pesticide residue grade or equivalent.

5.10 Carbitol: 2-(2-Ethoxyethoxy) ethanol

5.11 Hexane: pesticide residue grade or equivalent

5.12 Isooctane: pesticide residue grade or equivalent

5.13 Organic-free reagent water.

5.14 1+9 Hydrochloric Acid: For rinsing glassware (1 volume of HCL and 9 volumes of reagent water).

5.15 Silicic Acid: (H_2SIO_5) - 100 mesh powder.

5.16 10N Sodium Hydroxide

5.17 Sodium Chloride: (NaCl) - Pre-baked at 400°C for at least 4 hours.

5.18 Laboratory Reagent Grade Water

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Aqueous samples are collected in a 1L amber glass bottle. Samples are stored at 4 (±) °C until extraction.

Holding time for extraction of aqueous samples for Method 8151 is 7 days from date of sample collection, although the analyst should be aware that actual holding times employed may be project/program specific.

Store all extracts at 4°C (±2°C) in labeled Teflon-sealed containers. See SOP SD-902, "Sample Receipt and Internal Control," current revision, for storage areas and temperature maintenance procedures.

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR HERBICIDE ANALYSIS - METHOD 8151

7.0 PROCEDURES

INITIAL EXTRACTION

7.1 Thoroughly rinse all glassware and separatory funnels to be used in this procedure with 1+9 HCL, then Laboratory reagent grade water, acetone and methylene chloride. (Note: All glassware used between this step and the Diazomethane esterification must be pre-rinsed in this fashion.) Use only glassware that has been acid rinsed.

7.2 Assign a separate quality control number to each blank and associated spike and record this information in the appropriate log book. One blank and at least one LCS should be extracted per batch.

7.3 Sample specific matrix spikes and matrix spike duplicates are extracted per client request or per project requirement. When the client does not specify sample QC, then the extractions lab will chose a sample for quality control (one set per 20 samples designated as MS/MSD) to extract and analyze. If sufficient volume of sample(s) is not available for an MS, MSD, the lab will extract an LSC, LCSD instead.

7.4 Assemble, label, and methylene chloride rinse a 2 liter separatory funnel with stopcock and closure, a 500 mL glass sample bottle, and a 2 Liter Na_2SO_4 jar for each sample including blank, lab control sample and lab control sample duplicate. Make sure no residual $MeCl_2$ is present on glassware.

7.5 Determine sample bottle volumes by comparing to reference bottle. Record sample volumes in logbook. Transfer the 1-L sample aliquot to a 2-L separatory funnel. For TCLP samples, transfer 200ml of sample to funnel using a pre-rinsed 1000 mL graduated cylinder. 800mL DI water is added to all TCLP samples.

7.6 Laboratory reagent grade water will serve as the method blank and lab control sample (LCS). For each blank and LCS, add 1000 mL of DI water to the separatory funnel using a clean 1000 mL graduated cylinder. If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or group supervisor.

7.7 Add 250g of NaCl to the samples and the QC. Seal and shake to dissolve the salt.

7.8 Using a gas-tight volumetric syringe, add 1mL of herbicide surrogate solution to all of the samples, including blank and LCS's. Record surrogate number and amount added. Rinse syringes with acetone before and after use. Extreme accuracy should

be used when measuring and adding spike and surrogate solutions! Double check the solution number and amount used.

7.9 Add 1mL herbicide spiking solution to LCS and MS/MSD, as required. Record spike number and amount added in appropriate logbook. Rinse syringes with methanol before and after use.

Hydrolysis

7.10 Add 17 mL of 10N NaOH to all 1-L samples (3.4ml to TCLP samples), seal, and shake. Using pH paper, adjust the pH to 12 or greater by adding more 10N NaOH if necessary. Shake for 3 minutes on mechanical shaker. Let samples sit for 20 minutes, shake for 3 minutes and repeat three times. This will complete the hydrolysis step.

Solvent Washes

7.11 Add 60mL of methylene chloride to the sample bottles, rinse the bottles and add the rinses to the separatory funnel. Extract the sample by vigorously shaking the funnel for 3 minutes, with periodic venting to release excess pressure. Allow the organic layer and the water layer to separate for a minimum of 10 minutes. Discard the methylene chloride layer.

7.12 Repeat step 7.11 two more times, discarding the methylene chloride layer each time.

Extraction

7.13 Add 17 mL of cold (4°C) 12N sulfuric acid to all samples, seal, and shake to mix. Using pH paper, adjust the pH of the sample to 2 or less than 2 by adding more 12N sulfuric acid if necessary.

7.14 Add 120 mL ether to each separatory funnel. Shake and vent until there is no more pressure build-up. Shake on mechanical shaker for 3 minutes. Allow the phases to separate for 10 minutes.

7.15 Collect the aqueous (bottom) layer in a 2 liter Na_2SO_4 jar, and the ether (top) layer in a 500 mL sample bottle containing about 20g of acidified Na_2SO_4. Cap and shake the ether layer and drying agent. Return the aqueous layer to the separatory funnel.

7.16 Extract the aqueous layer two more times with 60 mL aliquots of ether as in steps 7.14-7.15. Combine ether (top layer) in the sample bottle. Prior to last extraction, rinse the 2-L Na2SO4 jar with ether and transfer to funnel to remove any remaining analytes.

7.17 Dispose the aqueous layer in the "N low" waste (sep-funnel waste) container. To minimize ether exposure in the laboratory, allow glassware to air dry in a fume hood overnight or rinse them with tap water before bringing to dish washing area.

Drying Step

7.17 Additional acidified Na_2SO_4 is added to the extract if it is not free flowing crystals or it is in a cake form. Shake the extract (ether phase) and the drying agent for one minute.

7.18 Allow the extract to remain in contact with the Na_2SO_4 for at least two hours, but, preferably stored overnight in the fume hood.

Note: The drying step is very critical to ensuring complete esterification. Any moisture remaining in the ether will result in low herbicide recoveries. The amount of sodium sulfate is adequate if some free flowing crystals are visible when swirling the flask. If all of the sodium sulfate solidifies in a cake, add a few additional grams of acidified sodium sulfate and again test by swirling. Neutralize the aqueous layer in a hood, then dispose in the Sep. Funnel waste container. To minimize ether exposure in the laboratory, allow glassware to air dry in a fume hood before bringing to dish washing area.

EXTRACT CONCENTRATION

7.21 Assemble a Kuderna-Danish apparatus with concentrator tube for each sample. Rinse the KD glassware with methylene chloride making sure no residual $MeCl_2$ is present on glassware before transferring samples.

7.22 Carefully, decant the extract into the K-D apparatus. Avoid allowing any acidified sodium sulfate crystals to fall in the concentration tube. Use a glass rod to crush any caked sodium sulfate in the glass jar. Rinse the glass jar 3 times with 10 mL of ether. Let drain between rinses. Thoroughly rinse funnel with ether and let drain.

7.23 Add 2 clean boiling chips to the K-D collector and attach a Macro-snyder column. Pre-wet the column with ether and place the K-D apparatus on the steam bath (which is heated no higher than 60°C). When the volume of liquid reaches approximately 2-4 mL, remove from the steam bath and allow to drain and cool for several minutes. Use caution, the ether will evaporate rapidly!

7.24 Remove the column and rinse the flask and its lower joint into the concentrator tube with 1 to 2 mL of ethyl ether.

7.25 Concentrate the sample to 0.5 mL using the nitrogen blow-down apparatus.

7.26 Add 1.0 mL of isooctane and 0.5 mL of methanol. Rinse pipette and sides of tube to dilute to final volume of 4.0 mL with ether.

DIAZOMETHANE ESTERIFICATION

VERY IMPORTANT: Diazomethane is a toxic carcinogen and can explode under certain conditions! The following precautions MUST be followed:

- Use only a well ventilated hood. Do not breathe the vapors.
- Do not heat above 90□C. Explosion may result.
- Diazomethane must NEVER come into contact with ground glass surfaces as rough surfaces are proven initiators of detonations.
- Avoid exposure of the solution to bright light - explosion may result.
- Always use EXTREME caution when handling either diazomethane or Diazald.

7.27 Assemble the diazomethane bubbler (see Figure 2).

7.28 Add 5.0 mL of ether to tube I. Add about 2.0-3.0 mLs of the Diazald solution (from 5.6) and 1.5 mL of 37% KOH to tube 2. Add the 37% KOH solution last to begin the reaction, and quickly cap both tubes.

7.29 Apply a nitrogen flow of 5-10 mL/min so bubbles emerge slowly. Bubble directly into the sample extract's concentrator tube. Allow the diazomethane to bubble through the sample for 1-2 minutes or until the yellow color persists. There is sufficient Diazald solution for esterification of two, maybe three, samples. Rinse exit tube with ether between samples.

7.30 Remove the exit tube form the KD concentrator tube, cover and store at room temperature for 20-30 minutes.

7.31 Destroy any unreacted diazomethane by adding 0.1 to 0.2 g silicic acid to the collectors. Allow to stand until the evolution of nitrogen is complete. Pipette the extract into a hexane rinsed 12 mL vial and rinse the collector twice with hexane (1-2 mL each time). Be careful to leave the silicic acid in the concentrator tubes. Bring to a final volume of 10mL using a reference vial, mix well, and let sit for a few minutes.

7.32 The extract is now ready for analysis. Make sure that initial volumes, intermediate volumes, aliquot volumes, and final volumes have been recorded in the logbook.

Also verify that the surrogate and spike identification numbers and amounts used have been recorded as well as the Diazald identification number. The temperature of the water in the nitrogen evaporation water bath is recorded in the extraction logbook. The lot numbers of all of the solvents, acids and bases, sodium sulfate, sodium chloride, as well as all of the filter papers that are used in the extraction process are recorded in the logbook. Any deviations from the SOP or any abnormal sample observation should be noted as comments.

7.33 The data entered here are later used in calculations of the final result; see SOP CA-305, Analysis of Herbicides in Extracts of Water & Soil.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

8.1 The purity of the solvents used is checked on a lot number basis and is kept on file in the Extractions Laboratory.

8.2 A method blank and a laboratory control sample (LCS) must be extracted for each and every item listed below:

- Each sample matrix (water)
- Each extraction method or level
- Every batch of 20 samples, or fewer, extracted in a 24-hour period

8.3 A matrix spike (MS), and matrix spike duplicate (MSD) should be prepared every 20 samples.

8.4 Sample specific matrix spikes and matrix spike duplicates are extracted per client request or per project requirements. When the client does not specify sample QC, the extractions lab will choose one (per 20) samples for quality control to extract and analyze.

8.5 Surrogate and Spike acceptability criteria can be found in SOP CA-305, Analysis Of Chlorinated Herbicides by GC Using Methylation Derivatization: SW-846 Method 8151.

8.6 If all quality control criteria are not met, appropriate steps must be taken to determine the cause. Problems indicate either matrix effect or an out of control event in the procedure.

8.7 Batch QC Requirements: If surrogates or spike compounds fail their criteria in the blank or lab control samples, the entire extraction batch is in question. A Corrective Action Report is initiated in the GC lab, and completed using information obtained from the Organics Prep Lab. If possible, the entire batch of samples is re-extracted with new QC samples. If no more sample can be obtained for re-extraction, any

results reported must be flagged in the report and a narrative is included qualifying the data. Refer to SOP CA-305, Analysis of Chlorinated Herbicides by GC Using Methylation Derivatization: SW-846 Method 8151, for further details.

Each extractions analyst must demonstrate proficiency in performing the extractions that prepare samples for analysis. Demonstration consists of preparation (extraction), by the analyst, of at least four aliquots which are then analyzed according to the analytical method in question. These QC samples must meet all quality control acceptance limits. Demonstration must be documented by use of a form which summarizes the results of the analysis of these aliquots, calculated percent recoveries, and standard deviation. Demonstration of proficiency must be done one time per analyst initially and then annually thereafter. Refer to SOPs QA-805 and QA-807, current revision.

If, upon analysis of the extracted samples, it is discovered that quality control acceptance criteria have not been met, all associated samples must be evaluated against all the QC. In some cases data may be reported, perhaps with narration, while in other cases, other corrective action may be taken. The corrective actions may include re-extraction of the samples associated with the quality control sample that did not meet acceptance criteria, or may include making new reagents and standards if the standardization is suspect. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases “qualified” data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Organic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit and Instrument Detection Limit Studies, for procedures on determining the MDL.

Refer to the applicable analytical SOP for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

"Test Methods for the Evaluation of Solid Waste: Physical/Chemical Methods", SW-846, third Edition, Final Update III, December 1996, Method 8151A.

Katahdin SOP CA-305, Analysis of Chlorinated Herbicides by GC Using Methylation Derivatization: SW-846 Method 8151, current revision.

LIST OF TABLES AND FIGURES

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR HERBICIDE ANALYSIS - METHOD 8151

TABLE 1

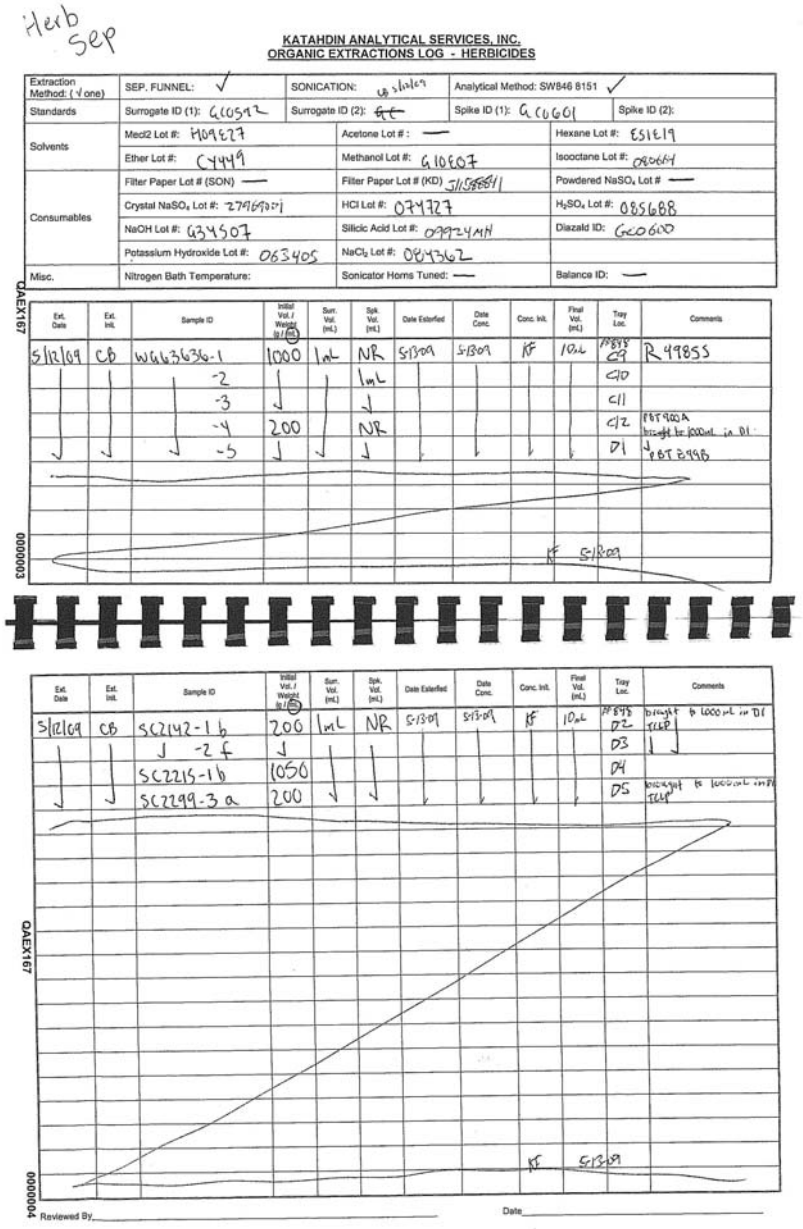
SUMMARY OF METHOD MODIFICATIONS

Topic	Katahdin SOP CA-516-05	Method 8151, current revision
Apparatus/Materials	Use nitrogen blowdown technique.	Use two ball micro Snyder column.
Reagents	Unpreserved diethyl ether. 10N NaOH. Acidify sulfate in Hexane.	Ethyl ether preserved with BHT. 6N NaOH. Acidify sulfate in Ether.
Sample preservation/ handling		
Procedures	Shake for 3 minutes. Samples poured through acidified sulfate before KD.	Shake for 2 minutes. Samples poured through acidified glass wool before KD.
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR HERBICIDE ANALYSIS - METHOD 8151

FIGURE 1

EXAMPLE OF AQUEOUS HERBICIDE LOGBOOK PAGE



Herb Sep

KATAHDIN ANALYTICAL SERVICES, INC.
ORGANIC EXTRACTIONS LOG - HERBICIDES

Extraction Method: (√ one)	SEP. FUNNEL: √	SONICATION:	Analytical Method: SW846 8151 √
Standards	Surrogate ID (1): GC0592	Surrogate ID (2): ~~GC~~	Spike ID (1): GCUG01 / Spike ID (2):
Solvents	MeCl2 Lot #: H09627	Acetone Lot # : —	Hexane Lot #: E51E19
	Ether Lot #: C4449	Methanol Lot #: G10E07	Isooctane Lot #: 080664
Consumables	Filter Paper Lot # (SON) —	Filter Paper Lot # (KD) J1558841	Powdered $NaSO_4$, Lot # —
	Crystal $NaSO_4$ Lot #: 27969201	HCl Lot #: 074727	H_2SO_4 Lot #: 085688
	NaOH Lot #: 634507	Silicic Acid Lot #: 09924MH	Diazald ID: GC0600
	Potassium Hydroxide Lot #: 063405	$NaCl_2$ Lot #: 084362	
Misc.	Nitrogen Bath Temperature:	Sonicator Horns Tuned: —	Balance ID: —

QAEX167

Ext. Date	Ext. Init.	Sample ID	Initial Vol. / Weight (g/mL)	Surr. Vol. (mL)	Spk. Vol. (mL)	Date Extarfied	Date Conc.	Conc. Init.	Final Vol. (mL)	Tray Loc.	Comments
5/12/09	CB	WG63636-1	1000	1mL	NR	5/13/09	5/13/09	KF	10mL	PBT 8995 C9	R49855
		-2			1mL					C10	
		-3								C11	
		-4	200		NR					C12	PBT 8994 brought to 1000mL in DI
		-5								D1	PBT 8996

0000003

Ext. Date	Ext. Init.	Sample ID	Initial Vol. / Weight (g/mL)	Surr. Vol. (mL)	Spk. Vol. (mL)	Date Extarfied	Date Conc.	Conc. Init.	Final Vol. (mL)	Tray Loc.	Comments
5/12/09	CB	SC2142-1b	200	1mL	NR	5/13/09	5/13/09	KF	10mL	PBT 8995 D2	brought to 1000mL in DI TCLP
		-2f								D3	
		SC2215-1b	1050							D4	
		SC2299-3a	200							D5	brought to 1000mL in DI TCLP

QAEX167

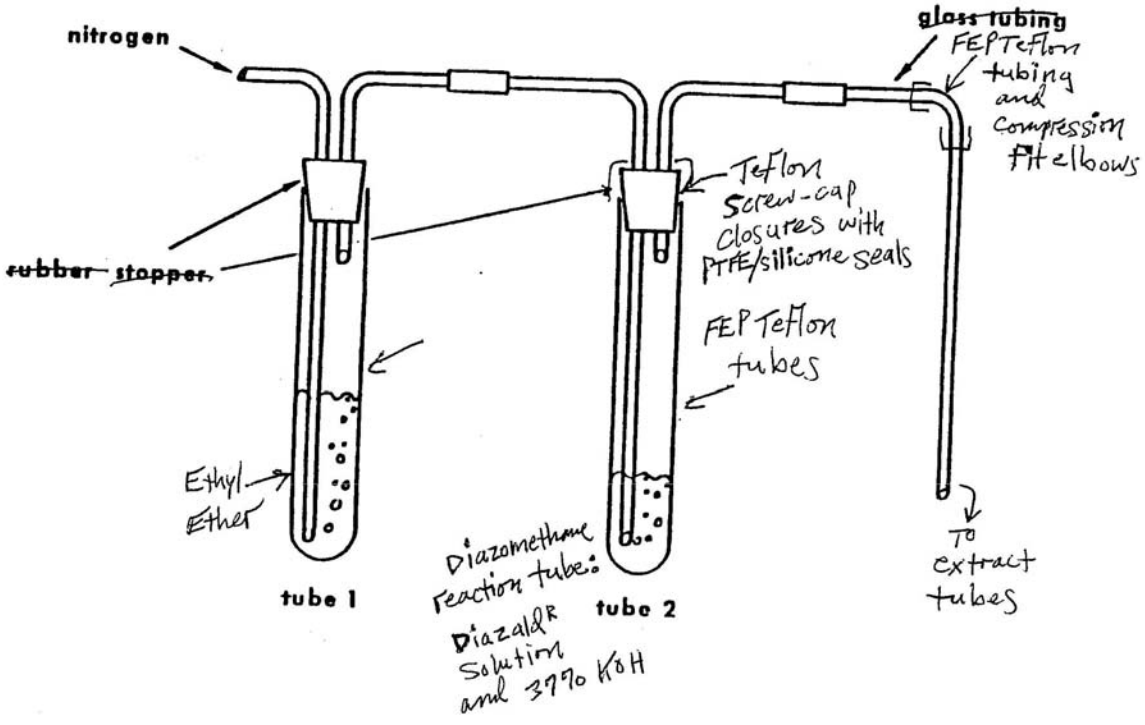
0000004

Reviewed By_____ Date_____

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR HERBICIDE ANALYSIS - METHOD 8151

FIGURE 2

DIAZOMETHANE SOLUTION GENERATOR



TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS

Prepared By: Mike Thomas Date: 7/98

Approved By:

Group Supervisor: Michael F. Thomas Date: 11/15/00

Operations Manager: [signature] Date: 11/15/00

QA Officer: Deborah J. Nadeau Date: 11·15·00

General Manager: [signature] Date: 11/16/00

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Minor changes throughout. Clarifications to procedure section.	DN	11·15·00	11/15/00
02	Added definitions to section 1.1. Wording changed or added to clarify sections 5, 6, 8, + 9. New figure	MRC	11.08.04	11.08.04
03	Sect. 7.1.2 - adding the step to rinse forceps also. 7.10 adding condenser temperature and output voltage of variable transformer	LAD	04/06	04/06
04	Added generated waste information. Updated spike list. Added LCSD. Reworded Sect. 7.10 and 7.11 for clarification. Updated Table 1 Replaced figure 1	LAD	09/07	09/07
05	Changed "N-Lo" waste to "K" waste. Updated Logbook example. Sect. 7 - added wording instructing the recording of consumable lot #'s in Logbook.	LAD	07/08	07/08

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Added balance criteria. Changed weight criteria to +/- 0.05g. Minor changes to section 7 to reflect current techniques. Clarified samples being GPC'd are not solvent exchanged into hexane. Updated logbook page. Added CA-108 reference for subsampling information	LAD	08/09	08/09

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-524-06**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-524-06**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS.**

Recipient: _____ Date: _____

1.0 SCOPE AND APPLICATION

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

The purpose of this SOP is to describe the procedure for extracting pesticides/PCBs from solids such as soils, sludges, and wastes using Method 3540. The Soxhlet extraction process ensures intimate contact of the sample matrix with the extraction solvent.

This method is applicable to the isolation and concentration of water-insoluble and slightly water-soluble organics in preparation for a variety of chromatographic procedures including methods 8081 for pesticides and 8082 for PCB's.

1.1 Definitions

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the extraction of samples for pesticide/PCB analysis. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training and Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in the extraction of

samples for pesticide/PCB analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to indicate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Waste Disposal

Wastes generated during the preparation of samples must be disposed of in accordance with the procedures described in the current revision of the Katahdin Hazardous Waste Plan and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the rinsing of glassware etc. should be disposed of in the “D” waste stream satellite accumulation area nearest the point of generation. Acetone and hexane are considered flammable waste, and should be disposed of in the “O” waste stream satellite accumulation area nearest the point of generation. Post-extraction soil samples, used glass wool, and sodium sulfate waste should be disposed of in the soil with organics “I” waste stream satellite accumulation area nearest the point of generation. Acid waste generated

during the cleanup of PCB samples should be disposed of in the "K" satellite accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

2.0 SUMMARY OF METHOD

2.1 The solid sample is mixed with anhydrous sodium sulfate, placed in a Soxhlet extractor and extracted with methylene chloride.

2.2 The extract is then dried, concentrated, and exchanged into hexane for GC analysis. Sulfuric acid cleanup is performed on extracts for 8082 PCB analysis.

3.0 INTERFERENCES

Solvents, reagents, glassware, and other sample preparation apparatus may yield interferences to GC analysis due to the presence of contaminants. These contaminants can lead to discrete artifacts or elevated baselines in chromatograms. Routinely, all of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running reagent blanks. Interferences caused by phthalate esters can pose a major problem in pesticide analysis. Common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory. At no time may gloves that have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Additionally, whenever possible plastic items in this lab must be replaced with metal or teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure clean glassware and apparatus are used, pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis.

Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to inteferences, further cleanup of the sample extract may be needed to minimize interferences.

4.0 APPARATUS AND MATERIALS

4.1 a) Soxhlet extractor – 45/50 top joint and 24/40 lower joint.

b) 500 mL flat-bottom boiling flask

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

c) Allihn cooling water condenser

4.2 Powder Funnels – 100 mm top diameter, 35 mm stem

4.3 Kuderna-Danish (K-D) apparatus

4.3.1 Concentrator tube - 10-mL

4.3.2 Evaporation flask - 500-mL

4.3.3 Snyder column - Three-ball macro

4.4 Nitrogen evaporation (N-EVAP) apparatus.

4.5 Boiling stones, 12 mesh silicon carbide (carborundum) – pre-purified by Soxhlet extraction in methylene chloride

4.6 Water bath - Heated, with concentric ring cover, capable of temperature control (± 5°C). The bath should be used in a hood.

4.7 Vials - Glass, 4, 12, or 16 mL with Teflon-lined screw caps

4.8 Glass wool (fiberglass) - baked at 400°C for a minimum of 4 hours or overnight.

4.9 Heating mantles - Rheostat controlled.

4.10 Disposable glass Pasteur pipets, 5 ¾", and bulbs.

4.11 Drying oven - capable of maintaining 105°C for glassware drying.

4.12 Muffle oven – capable of maintaining 400 °C for baking glass wool and organic-free sand.

4.13 Beakers, 250 or 400 mL

4.14 Top-loading balance - capable of weighing to 0.**01** g.

4.15 Spatulas, stainless-steel

4.16 Long forceps, stainless-steel

4.17 Metal clips – for securing Soxhlets to boiling flasks

4.18 Filter paper, 18.5 cm, Fisherbrand or Whatman 114V (or equivalent)

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

5.0 REAGENTS

5.1 Sodium sulfate (granular, anhydrous and powdered, anhydrous) (ACS reagent grade), Na_2SO_4. Certified by the manufacturer/vendor as purified by heating at 400°C for 4 hours prior to receipt by the laboratory.

5.2 Methylene chloride - (pesticide grade or equivalent) purchased by lot, evaluated prior to use by concentration of 300 mL to 1 mL followed by GC/MS analysis.

5.3 Acetone and hexane – (pesticide grade or equivalent) purchased by lot, evaluated prior to use by concentration of 300 mL to 1 mL followed by GC/MS and GC analysis.

5.4 Organic-free sand, purified by baking at 400 °C at a minimum of 4 hours or overnight. Method blanks serve as checks on the baked sand.

5.5 Surrogate spiking solution - Prepare a solution of decachlorobiphenyl (DCB) and tetrachloro-meta-xylene (TCMX) at a concentration of 1 ug/mL in acetone.

5.6 Matrix Spike/Lab Control Sample spiking solution

5.6.1 Pesticide spike solution – prepare in pesticide grade methanol containing the analytes listed below at concentrations of 0.5 ug/mL.

4,4'-DDD	Endrin
4,4'-DDE	Endrin Aldehyde
4,4'-DDT	Endrin Ketone
Aldrin	gamma-BHC (Lindane)
alpha-BHC	Heptachlor
beta-BHC	Heptachlor Epoxide
delta-BHC	Methoxychlor
Dieldrin	alpha-Chlordane
Endosulfan I	gamma-chlordane
Endosulfan II	Endrin
Endosulfan Sulfate	Endrin Aldehyde

5.6.2 PCB spike solution – prepare Aroclor 1660 (Aroclor 1016 and 1260) in pesticide grade acetone at a concentration of 5.0 ug/mL each.

5.7 Store the solutions mentioned in sections 5.5 and 5.6 at -10 to -20 °C (±2 °C) in a Teflon sealed container. Solution must be verified by GC/ECD prior to use and must be replaced every 6 months or sooner if degradation is evident.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

Sediment/soil samples must be collected in a soil jar and must be maintained at 4°C (±2°C).

Holding time for extraction of sediment/soil samples for Method 3540 is 14 days from date of sample collection, although the analyst should be aware that actual holding times employed may be project/program specific.

Store all extracts at 4°C (±2°C) in the dark in labeled Teflon-sealed containers. See SOP SD-902, "Sample Receipt and Internal Control," current revision, for storage areas and temperature maintenance procedures.

7.0 PROCEDURES

7.1 Preparing the Soxhlet Extraction Apparatus

7.1.1 Rinse the Soxhlet extractors and 500 mL flat-bottom boiling flasks three times with methylene chloride. Be sure that the solvent rinses through the large vapor tube and smaller siphon tubes of the Soxhlet. Inspect these for tiny cracks. Also rinse the 24/40 lower joint.

7.1.2 Add ~ 250 mLs of methylene chloride to the 500 mL boiling flask. Add several boiling stones. Rinse the stainless steel forceps and pre-baked glass wool with Methylene chloride. Working in a hood, place a plug of the glass wool at the bottom of the Soxhlet so that the siphon tube hole is covered. Insert the 24/40 joint of the Soxhlet extractor into the 500 mL boiling flask and secure with a metal clip. Cover the top of the Soxhlet extractor with a piece of aluminum foil until ready to begin loading the sample.

7.2 Sample Handling

7.2.1 Sediment/soil samples - Decant and discard any water layer on a sediment sample. Mix the sample thoroughly with the stainless steel spatula. If the sample container is full to the extent that stirring the sample is impractical, try to remove the “best representative” aliquot from the jar based on color, particle size, moisture, etc. Discard any foreign objects such as sticks, leaves, and rocks.

7.2.2 Gummy, fibrous, or oily materials not amenable to mixing should be cut, shredded, or otherwise reduced in size to allow for maximum exposure of the sample surfaces to the extraction solvent. Materials such as glass, rubber, metal, etc. may not require mixing with powdered sodium sulfate to disperse the sample. Plastic materials must be tested for degradation (melting) in methylene chloride prior to Soxhlet extraction.

7.2.3 Please refer to Katahdin Analytical Services SOP CA-108, current revision, "Basic Laboratory Techniques" for more information of subsampling.

7.3 Weigh out a 30.00 ± 0.05 g portion of sample into a labeled 400 mL beaker. Record sample weight to nearest 0.05 g in appropriate extraction logbook. Add between 30 to 60 g of powdered sodium sulfate, as required, to produce a "free-flowing" mixture. The amount of sodium sulfate added will depend upon the moisture content of the sample (e.g., low moisture content will require less sodium sulfate). Mix well with a spatula. Keep the spatula in the sample beaker and cover the beaker with aluminum foil. Record sodium sulfate lot in logbook.

7.4 A method blank must be prepared with each extraction batch, not to exceed 20 client samples. To prepare method blank, weigh out one 30.0 ± 0.05 g portion of purified sand in a labeled 400 mL beaker. Record sample weights to nearest 0.05 g in appropriate extraction logbook. Add 60 g sodium sulfate and mix well. (Although a "free-flowing" mixture can be achieved with less than 60 g sodium sulfate, the method blank must contain 60 g in order to evaluate the sodium sulfate as a potential source of contamination.)

7.5 A laboratory control sample (LCS) must be prepared with each extraction batch, not to exceed 20 client samples. To prepare LCS, weigh out one 30.0 ± 0.05 g portion of purified sand in a labeled 400 mL beaker. Record sample weights to nearest 0.05 g in appropriate extraction logbook. Add 30 g sodium sulfate and mix well. With extraction batches prepared for combined 8081/8082 Pesticide and PCB analysis, separate Pesticide and PCB LCS's must be prepared (refer to section 5.6). If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or Department Manager.

7.6 A matrix spike/matrix spike duplicate (MS/MSD) set must be prepared for every 20 samples. To prepare MS/MSD, weigh out 30.0 ± 0.05 g portions of the sample designated for MS/MSD into each of two labeled 400 mL beakers. Record sample weights to nearest 0.05 g in appropriate extraction logbook. Add 30 g sodium sulfate to each to produce a free-flowing mixture, and mix well. With extraction batches prepared for combined 8081/8082 Pesticide and PCB analysis, separate Pesticide and PCB MS/MSD pairs must be prepared (refer to section 5.6).

7.7 Once all of the QC and field samples have been weighed and mixed with sodium sulfate, begin adding each to the assembled and appropriately labeled Soxhlet extractors using the stainless steel spatulas. Carefully scrape all of the mixtures from the beaker walls so that no more than 1% remains behind in the beaker. Be careful not to have any of the solid material fall into the extract flask through the large vapor tube.

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7.8 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL of the pesticide/PCBs surrogate spiking solution using a 1.0 mL gas tight syringe. Record surrogate spike volume and identification code in the extraction logbook. Thoroughly rinse syringe with solvent between each use.

7.9 To LCS/LCSD and MS/MSD add 1.0 mL of either the pesticide or PCBs matrix spike/LCS spiking solutions using a 1.0 mL gas tight syringe. Record matrix spike/LCS spiking solution volume and identification codes in the extraction logbook. Thoroughly rinse syringe with solvent between each use.

7.10 Rinse the joints of the Allihn cooling condensers with Methylene Chloride, collecting the waste in a methylene chloride solvent waste container. Place each of the Soxhlet extractors in a heating mantle and lower the Allihn cooling water condensers into the 45/50 joints of the extractors. The condensers should be set to a temperature of 15ºC. Save the pieces of aluminum foil for covering the Soxhlets when the extraction is complete. Switch on the individual heating mantles and be sure that the Rheostat of the variable transformer is set to 55% of the output voltage. Once the methylene chloride begins to boil and the Soxhlet begins to cycle (solvent will immerse the sample and collect in the Soxhlet until the level reaches that of the small siphon tube and then begin to spill over into the extract flask), re-check the apparatus' for leaks. Allow the samples to extract for 18-24 hours. Be sure the chiller/recirculator temperature is set low enough to provide enough cooling capacity for the number of extractions in the batch.

7.11 When the extraction is complete, allow the extracts to cool before dismantling. Remove the Allihn condenser and replace the aluminum foil on top of the extractor. Move the extractors to a hood and detach the extractor from the extract flask. Tilt each extractor slightly to cause any remaining solvent in the sample chamber to drain through the siphon tube into the extract flask. This will help to cool the extract flask and make the apparatus easier to dismantle. Try to drain as much solvent as possible from the extractor into the flask. **This is done by rinsing a glass tube in methylene chloride and pressing on the sample slightly so that as solvent as possible is drained into the extract flask.** Cover the flask with aluminum foil and store in the interim extract storage refrigerator unless the extracts are to be concentrated the same day.

7.12 Immediately remove the extracted soil/sodium sulfate mixtures from the extractors using a square edge spatula, and dispose of in an appropriate solid waste container. It is important to do this soon after the extractors are dismantled, as the sample mixture will tend to "freeze" into a solid mass in the Soxhlet as the solvent dries.

CONCENTRATION OF THE EXTRACTS

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

7.13 Rinse the K-D glassware (flask, concentration tube, and Snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride before assembling. Add a few boiling chips to the K-D. Insert fluted 18.5 cm filter papers into short stem powder funnels and add ~ 2 inches of sodium sulfate crystals. Rinse the sodium sulfate in the assembled funnels with ~20-30 mLs of methylene chloride. Place the assembled K-D's under the funnels. Record the lot numbers of the solvent, sodium sulfate and filter papers in the extraction logbook.

7.14 If samples are to be GPC'd, refer to the current revision of Katahdin SOP CA-513, Extract Cleanup Using Gel Permeation Chromatography, for appropriate concentrating procedures. Samples that undergo GPC are not solvent exchanged into hexane.

7.15 If samples are not to be GPC'd follow Steps 7.16 through 7.23 to concentrate extracts to final volume of 10.0 mLs

7.16 For a solvent exchange, (for samples not being GPC'd), add approximately 50 mL hexane to funnel and let drain through. Since methylene chloride has a lower boiling point than Hexane, this will result in a final extract in hexane only. Record the lot number of the solvent in the extraction logbook.

7.17 Transfer the extracts to the K-D concentrator setups through the sodium sulfate in the funnels. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to pouring through the sodium sulfate. After pouring all of the extract volume through the sodium sulfate, rinse the extract flask three times with ~ 2 – 3 mLs of methylene chloride. Add the rinsings through the sodium sulfate to complete a quantitative transfer. Rinse the sodium sulfate with ~ 15 mLs of methylene chloride and allow to drain.

7.18 Transfer the labels from the extract flasks to the K-Ds. Remove the funnel and attach a 3-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride.

7.19 Place the K-D in a hot water bath (75-85°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 6 mL, remove the K-D from the water bath. Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of methylene chloride, hexane, if exchange is taking place. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask.

Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL methylene chloride, hexane, if exchange is taking place.

7.20 Reduce the extract in the concentrator tube to approximately 1-2 mL using the nitrogen blow-down apparatus to ensure all methylene chloride has been evaporated. The bath temperature must be no higher than the boiling point of the solvent (39°C for methylene chloride). Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with ≈1 mL of methylene chloride. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N_2 sparging pipet closer to the surface of the extract to expedite the concentration. Record the temperature of the water in the nitrogen evaporation water bath in the extraction logbook also note any problems or extract losses, if they occur..

7.21 Complete quantitative transfer of the extract to a vial by using hexane. Adjust the volume of the hexane extract to 10 mL in either a 12 or 16 mL vial using the appropriate “reference vial” for volume comparison.

7.22 Label the vial with lab sample number, extraction date, matrix and analysis. Store extract vials at a temperature of 4 ± 2 °C until ready for analysis. Indicate in the extractions logbook the box number and “tray location” of the individual extract vials.

7.23 All sample extracts for 8082 PCB analysis must undergo a sulfuric acid wash (cleanup) prior to analysis, unless it has been GPC’d. All sample extracts for 8081 pesticide analysis should undergo further cleanup using the GPC unless time is a factor. All sample extracts for combined 8081/8082 analyses must be split unless GPC’d. One portion must be acid cleaned for 8082 analysis. The associated method blank must be split and acid-cleaned in the same fashion. PCB LCSs and matrix spikes are acid cleaned also. Pesticide LCSs and matrix spikes are not subjected to further cleanup. Record the lot number of the acid in the extraction logbook. Please refer to Katahdin SOP CA525 (current revision), Extract Cleanup Using Sulfuric Acid, for further instructions.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

A method blank must be extracted for each and every item listed below:

- Each sample matrix (soil, water)
- Each day of extraction (24 hours midnight - midnight)
- Each extraction method or level
- Every 20 samples extracted in a 24-hour period

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

A laboratory control sample (LCS) is required for each and every item listed below:

- Each sample matrix
- Each extraction method or level
- Every extraction batch of twenty or fewer samples
- Each analysis (pesticide and/or PCB) to be performed

Refer to the current revision of the applicable Katahdin SOP for analysis of Pesticides and PCBs for quality control acceptance criteria.

Each extractions analyst must demonstrate proficiency in performing the extractions that prepare samples for analysis. Demonstration consists of preparation (extraction), by the analyst, of at least four aliquots which are then analyzed according to the analytical method in question. These QC samples must meet all quality control acceptance limits. Demonstration must be documented by use of a form which summarizes the results of the analysis of these aliquots, calculated percent recoveries, and standard deviation. Demonstration of proficiency must be done one time per analyst initially and then annually thereafter. Refer to SOPs QA-805 and QA-807, current revision.

If, upon analysis of the extracted samples, it is discovered that quality control acceptance criteria have not been met, all associated samples must be evaluated against all the QC. In some cases data may be reported, perhaps with narration, while in other cases, other corrective action may be taken. The corrective actions may include re-extraction of the samples associated with the quality control sample that did not meet acceptance criteria, or may include making new reagents and standards if the standardization is suspect. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases “qualified” data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

9.0 METHOD PERFORMANCE

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Organic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Method Detection Limit, Instrument Detection Limit and reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the applicable analytical SOP for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste-Physical/Chemical Methods, Method 3540C, SW-846, Third Edition, Updates I, II, IIA, IIB, and III Revised December 1996, US EPA.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.1, 04/22/09.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

LIST OF TABLES AND FIGURES

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

TABLE 1

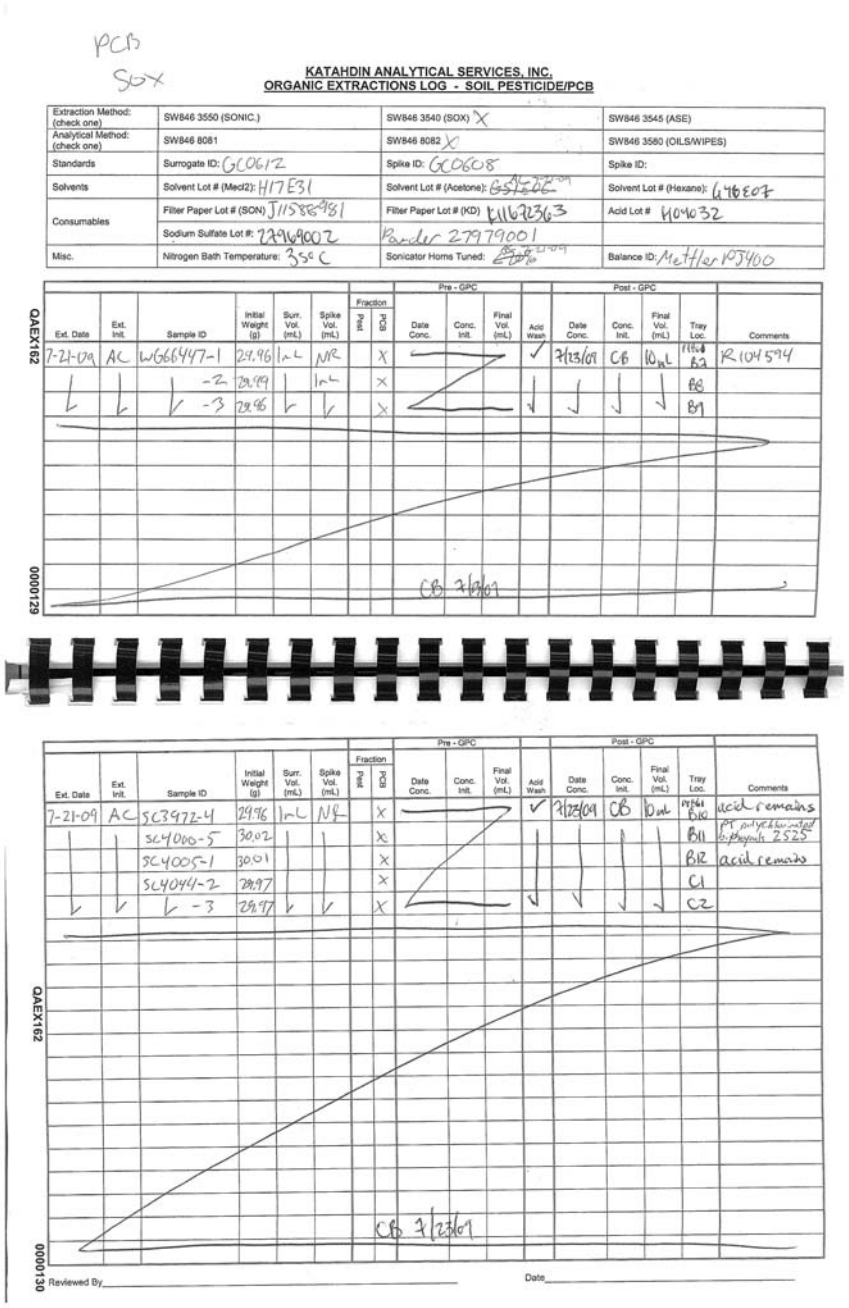
SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-524-06	METHOD 3540, current revision
Apparatus/Materials	1. short stem funnels	1. drying columns
Reagents		
Sample preservation/ handling		
Procedures	1. Use 30 grams of sample and 30 grams of sodium sulfate. 2. Use 250 mL of methylene chloride 3. no apparatus height specification for concentration on water bath 4. water bath at 75-85 deg C 5. sample removed from water bath when volume reaches ~6 mL 6. Solvent exchange to hexane is performed using K-D apparatus with addition of approximately 50 mL hexane at the start of concentration process	1. Use 10 grams of sample and 10 grams of sodium sulfate. 2. Use 300 mL of methylene chloride 3. partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-15min 4. water bath at 80-90 deg C 5. sample removed from water bath when volume reaches 1-2 mL 6. Solvent exchange to hexane is performed using K-D apparatus with addition of approximately 50 mL hexane after concentrating methylene chloride extract to 1 mL
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

FIGURE 1

EXAMPLE OF LOGBOOK PAGE



PCB
SOX

KATAHDIN ANALYTICAL SERVICES, INC.
ORGANIC EXTRACTIONS LOG - SOIL PESTICIDE/PCB

Extraction Method: (check one)	SW846 3550 (SONIC.)	SW846 3540 (SOX) X	SW846 3545 (ASE)
Analytical Method: (check one)	SW846 8081	SW846 8082 X	SW846 3580 (OILS/WIPES)
Standards	Surrogate ID: GC0612	Spike ID: GC0608	Spike ID:
Solvents	Solvent Lot # (MeCl2): H17E31	Solvent Lot # (Acetone): G5A226	Solvent Lot # (Hexane): 476E07
Consumables	Filter Paper Lot # (SON) J115886981	Filter Paper Lot # (KD) K1162363	Acid Lot # H04032
	Sodium Sulfate Lot #: 27969002	Powder 27979001	
Misc.	Nitrogen Bath Temperature: 35°C	Sonicator Horns Tuned:	Balance ID: Mettler PJ400

Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spike Vol. (mL)	Fraction: Petr	Fraction: PCB	Pre-GPC: Date Conc.	Pre-GPC: Conc. Init.	Pre-GPC: Final Vol. (mL)	Acid Wash	Post-GPC: Date Conc.	Post-GPC: Conc. Init.	Post-GPC: Final Vol. (mL)	Tray Loc.	Comments
7-21-09	AC	WG66447-1	29.96	1mL	NR		X				✓	7/23/09	CB	10mL	B2	R104594
↓	↓	↓ -2	29.99	↓	1mL		X				↓	↓	↓	↓	A8	
↓	↓	↓ -3	29.96	↓	↓		X				↓	↓	↓	↓	B9	

QAEX162 0000129

CB 7/23/09

Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spike Vol. (mL)	Fraction: Petr	Fraction: PCB	Pre-GPC: Date Conc.	Pre-GPC: Conc. Init.	Pre-GPC: Final Vol. (mL)	Acid Wash	Post-GPC: Date Conc.	Post-GPC: Conc. Init.	Post-GPC: Final Vol. (mL)	Tray Loc.	Comments
7-21-09	AC	SC3972-4	29.96	1mL	NR		X				✓	7/27/09	CB	10mL	A10 B10	acid remains
↓	↓	SC4006-5	30.02	↓	↓		X				↓	↓	↓	↓	B11	PT anthracene not in std 2525
↓	↓	SC4005-1	30.01	↓	↓		X				↓	↓	↓	↓	B12	acid remains
↓	↓	SC4044-2	29.97	↓	↓		X				↓	↓	↓	↓	C1	
↓	↓	↓ -3	29.97	↓	↓		X				↓	↓	↓	↓	C2	

QAEX162 0000130

CB 7/27/09

Reviewed By_____ Date_____

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS

Prepared By: Mike Thomas Date: 7/98

Approved By:

Group Supervisor: Michael Thomas Date: 11/15/00

Operations Manager: [signature] Date: 11/15/00

QA Officer: Deborah J. Nadeau Date: 11·16·00

General Manager: [signature] Date: 11/20/00

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Minor changes throughout. Clarifications to procedure section.	DN	11.16.00	11/16/00
02	Definitions added to section 1.1. Wording was added or changed to clarify sections 4, 5, 6, 7, 8 & 9. Minor changes throughout. New figures.	MRC	11.09.04	11.09.04
03 LAD 6-26-06	Updated Sect. 7.0 to include SIM. Updated figures 2 and 3 to include current SVOA compounds ~~MIXES~~ used. Updated Sect. 5.0 to include all compounds analyzed for. Updated logbook page. minor edits throughout.	LAD	04/06	04/06
04	Added waste generated information. Updated Spikes and Surrogates. Added SIM LCSD and MS/D requirements. Updated Table 1. Added GPC references. Added LCSD after LCS.	LAD	09/07	09/07
05	Updated logbook page. Added acidpate compounds to Fig. 2. Added recording of consomable's lot #'s and recording the Nitrogen water bath temp. in logbook	LAD	07/08	07/08

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Added balance criteria. Changed all weight criteria to +/- 0.05g. Revised section 7 to reflect current techniques. Added SOP CA-108 reference for sub-sampling. Updated logbook example	LAD	08/09	08/09

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

I acknowledge receipt of copy ___ of document **SOP CA-526-06**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-526-06**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS.**

Recipient: _____ Date: _____

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures and requirements for extracting semivolatile organic compounds from solids such as soils, sludges, and wastes using Method 3540. The Soxhlet extraction process ensures intimate contact the sample matrix with the extraction solvent.

This method is applicable to the isolation and concentration of water-insoluble and slightly water soluble organics in preparation for a variety of chromatographic procedures.

1.1 Definitions

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the extraction of samples for semivolatile analysis. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training and Documentation of Capability”.

It is the responsibility of all Katahdin technical personnel involved in the extraction of samples for semivolatile analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that the members of his/her group follow this SOP, to assure that their work is properly documented, and to indicate periodic review of the pertinent logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Waste Disposal

Wastes generated during the preparation of samples must be disposed of in accordance with the procedures described in the current revision of the Katahdin Hazardous Waste Plan and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the rinsing of glassware etc. should be disposed of in the “D” waste stream satellite accumulation area nearest the point of generation. Acetone and methanol are considered flammable waste, and should be disposed of in the “O” waste stream satellite accumulation area nearest the point of generation. Post-extraction soil samples, used glass wool, and sodium sulfate waste should be disposed of in the soil with organics “I” waste stream satellite

accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

2.0 SUMMARY OF METHOD

2.1 The solid sample is mixed with anhydrous sodium sulfate, placed in a Soxhlet extractor and extracted with methylene chloride.

2.2 The extract is then dried and concentrated for subsequent 8270 Semivolatile Organics analysis.

3.0 INTERFERENCES

Contaminants in solvents, reagents, glassware, and other sample processing hardware may cause method interferences such as discrete artifacts and/or elevated baselines in the total ion current profiles (TICPs). All of these materials routinely must be demonstrated to be free from interferences under the conditions of the analysis by running laboratory method blanks. Interferences caused by phthalate esters can pose a major problem in semivolatile organics analysis because many phthalates are also target analytes. Common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory. At no time may gloves that have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Additionally, whenever possible plastic items in this lab must be replaced with metal or Teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure clean glassware and apparatus are used, pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis.

Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to interferences, further cleanup of the sample extract may be needed to minimize interferences.

4.0 APPARATUS AND MATERIALS

4.1 Soxhlet apparatus:

a) Soxhlet extractor – 45/50 top joint and 24/40 lower joint.
b) 500 mL flat-bottom boiling flask

c) Allihn cooling water condenser

4.2 Powder Funnels – 100 mm top diameter, 35 mm stem

4.3 Kuderna-Danish (K-D) apparatus

4.3.1 Concentrator tube - 10-mL

4.3.2 Evaporation flask - 500-mL

4.3.3 Snyder column - Three-ball macro

4.4 Nitrogen evaporation (N-EVAP) apparatus.

4.5 Boiling stones, 12 mesh silicon carbide (carborundum) – pre-purified by Soxhlet extraction in methylene chloride

4.6 Water bath - Heated, with concentric ring cover, capable of temperature control (± 5°C). The bath should be used in a hood.

4.7 Vials - Glass, 1.8-mL capacity, with polytetrafluoroethylene (PTFE)-lined septum vials, and 12 mL with Teflon-lined caps for extracts designated for GPC cleanup.

4.8 Glass wool (fiberglass) - baked at 400°C for a minimum of 4 hours or overnight.

4.9 Heating mantles - Rheostat controlled.

4.10 Disposable glass pasteur pipets, 5 ¾" and bulbs.

4.11 Drying oven - capable of maintaining 105°C for glassware drying.

4.12 Muffle oven – capable of maintaining 400 °C for baking glass wool and organic-free sand.

4.13 Beakers, 250 or 400 mL

4.14 Top-loading balance - capable of weighing to 0.**01** g.

4.15 Spatulas, stainless-steel

4.16 Long forceps, stainless-steel

4.17 Metal clips – for securing Soxhlets to boiling flasks

4.18 Filter Paper, 18.5 cm, Fisherbrand or Whatman 114V (or equivalent)

5.0 REAGENTS

5.1 Sodium Sulfate - anhydrous powdered and granular crystals, reagent grade, certified by the manufacturer/vendor as purified heating to 400°C prior to receipt by the laboratory.

5.2 Methylene chloride, methanol, and acetone - pesticide residue analysis grade or equivalent. Methylene chloride and acetone are evaluated, by lot, prior to use, by concentration of approximately 400 mL to 1.0 mL followed by GC/MS analysis.

5.3 Organic-free sand, purified by baking at 400 °C. Method blanks serve as checks on the baked sand.

5.4 Base/Neutral and Acid (SVOA) Surrogate Spiking Solution - Surrogate standards are added to all samples and calibration solutions. Prepare a surrogate standard spiking solution that contains the following compounds at the indicated concentrations in acetone.

Compound	Conc.
phenol-$_{d6}$	100 ug/mL
2,4,6-tribromophenol	100 ug/mL
2-fluorophenol	100 ug/mL
nitrobenzene-$_{d5}$	50 ug/mL
p-terphenyl-$_{d14}$	50 ug/mL
2-fluorobiphenyl	50 ug/mL

Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem. If reanalysis of a method blank still indicates surrogates out of criteria, a new surrogate solution must be used.

5.5 SIM Surrogate Spiking Solution- Surrogate Standards are added to all samples and calibration solutions. Prepare a surrogate solution that contains the following compounds at a concentration of 2 ug/mL in acetone.

Compound	Conc. ug/mL
Fluorene-d10	2.0 ug/mL
2-Methylnaphthalene-d10	2.0 ug/mL
Pyrene-d10.	2.0 ug/mL
2,4-Dibromophenol	2.0 ug/mL

Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These

solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem. If reanalysis of a method blank still indicates surrogates out of criteria, a new surrogate solution must be used.

5.6 Base/Neutral and Acid (SVOA) Lab Control Sample / Matrix Spike Spiking Solution - Prepare a spiking solution in methanol that contains the following mixes listed in Figure 2 at a concentration of 50 ug/ml for the base/neutral compounds and 100 ug/ml for the acid compounds. Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

5.7 Base/Neutral (SIM) Matrix Spike/ Lab Control Sample Spike Solution for SIM-SVOA. Prepare a spiking solution in methanol that contains the compounds listed in Figure 2 at a concentration of 2 ug/mL for base/neutral. Take out 1.0 mL of Base/Neutral and Acid Matrix Spike/Lab Control Spiking Solution for SVOA and dilute it to 25.0 mL in methanol. Store the solution Spiking at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

5.8 Base/Neutral and Acid (SVOA) Appendix IX Lab Control Sample / Matrix Spike Spiking Solution – Prepare a spiking solution in methanol that contains the compounds listed in Figure 3 at concentrations of 100 ug/ml. Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Sediment/soil samples must be collected in a soil jar and must be maintained at 4°C (±2°C).

Holding time for extraction of sediment/soil samples for Method 3540 is 14 days from date of sample collection, although the analyst should be aware that actual holding times employed may be project/program specific.

Store all extracts at 4°C (±2°C) in the dark in labeled Teflon-sealed containers. See SOP SD-902, "Sample Receipt and Internal Control," current revision, for storage areas and temperature maintenance procedures.

7.0 PROCEDURES

All solid samples need to be cleaned up to reduce matrix interferences, time permitting. The cleanup procedure employed is gel permeation chromatography (GPC).

Sign chain-of-custody when removing and replacing samples in storage locations, and fill out the sample preparation/extraction log with the necessary information before starting the extraction. Prerinse all glassware three times with methylene chloride.

7.1 Preparing the Soxhlet Extraction Apparatus

7.1.1 Rinse the Soxhlet extractors and 500 mL flat-bottom boiling flasks three times with methylene chloride. Be sure that the solvent rinses through the large vapor tube and smaller siphon tubes of the Soxhlet. Inspect these for tiny cracks. Also rinse the 24/40 lower joint.

7.1.2 Add ~ 250 mLs of methylene chloride to the 500 mL boiling flask. Add several boiling stones. Using stainless steel forceps and working in a hood, place a plug of the pre-baked glass wool at the bottom of the Soxhlet so that the siphon tube hole is covered. Insert the 24/40 joint of the Soxhlet extractor into the 500 mL boiling flask and secure with a metal clip. Cover the top of the Soxhlet extractor with a piece of aluminum foil until ready to begin loading the sample. Record the solvent lot number in the extraction logbook.

7.2 Sample Handling

7.2.1 Sediment/soil samples - Decant and discard any water layer on a sediment sample. Mix the sample thoroughly with the stainless steel spatula. If the sample container is full to the extent that stirring the sample is impractical, try to remove the “best representative” aliquot from the jar based on color, particle size, moisture, etc. Discard any foreign objects such as sticks, leaves, and rocks.

7.2.2 Gummy, fibrous, or oily materials not amenable to mixing should be cut, shredded, or otherwise reduced in size to allow for maximum exposure of the sample surfaces to the extraction solvent. Materials such as glass, rubber, metal, etc. may not require mixing with powdered sodium sulfate to disperse the sample. Plastic materials must be tested for degradation (melting) in methylene chloride prior to Soxhlet extraction.

7.2.3 Refer to Katahdin SOP CA-108, current revision, “Basic Laboratory Technique” for more information on subsampling.

7.3 The following steps should be performed rapidly to avoid loss of the more volatile extractables. Weigh out a 30.00 $\pm$ 0.05 g portion of sample into a labeled 400-mL beaker. Record sample weight to the nearest 0.05 g in appropriate extraction logbook. Add between 30 g and 60 g of anhydrous powdered sodium sulfate as

required to produce a "free-flowing" mixture. The amount of sodium sulfate added will depend upon the moisture content of the sample (e.g., low moisture content will require less sodium sulfate). Mix well with a spatula. Keep the spatula in the sample beaker and cover the beaker with aluminum foil.

7.4 A method blank must be prepared with each extraction batch, not to exceed 20 client samples. To prepare method blank, weigh out one 30.00 ± 0.05 g portion of purified sand in a labeled 400 mL beaker. Add 60 g sodium sulfate and mix well. Although a "free-flowing" mixture can be achieved with less than 60 g sodium sulfate, the method blank must contain 60 g in order to evaluate the sodium sulfate as a potential source of contamination.

7.5 A laboratory control sample (LCS) must be prepared with each extraction batch, not to exceed 20 client samples. To prepare LCS, weigh out one 30.00 ± 0.05 g portion of purified sand in a labeled 400 mL beaker. Add 30 g sodium sulfate and mix well. If combined SIM-SVOA analysis is requested, a separate LCS must be prepared for each analysis. If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or Department Manager.

7.6 A matrix spike/matrix spike duplicate (MS/MSD) set must be prepared for every 20 samples. To prepare MS/MSD, weigh out 30.00 ± 0.05 g portions of the sample designated for MS/MSD into each of two labeled 400 mL beakers. Record sample weights to nearest 0.05 g in appropriate extraction logbook. Add 30 - 60 g sodium sulfate to each to produce a free-flowing mixture, and mix well. If combined SIM-SVOA analysis is requested, a separate MS/MSD must be prepared for each analysis.

7.7 Once all of the QC and field samples have been weighed and mixed with sodium sulfate, begin adding each to the assembled and appropriately labeled Soxhlet extractors using the stainless steel spatulas. Carefully scrape all of the mixtures from the beaker walls so that no more than 1% remains behind in the beaker. Be careful not to have any of the solid material fall into the extract flask through the large vapor tube.

7.8 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL of the appropriate base/neutral and acid surrogate spiking solution listed below using the pre-rinsed 1.0 mL gas tight syringe. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent prior to using it for another spiking solution.

7.8.1 If the request is for SVOA, use the SVOA surrogate solution (sect. 5.4)

7.8.2 If the request is for SIM, use the SIM surrogate solution (sect. 5.5).

7.8.3 If the request is for SIM-SVOA, use both the SIM and SVOA surrogate solutions. NOTE: Separate LCS/LCSD and/or MS/MSD are needed for each analysis and should be spiked with the appropriate surrogate.

7.9 To the LCS/LCSD and the MS/MSD add 1.0 mL of the appropriate base/neutral and acid (SVOA) matrix spike/LCS spiking solution listed below using a 1.0 mL gas tight syringe. Record matrix spike/LCS spiking solution volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent when spiking is completed.

7.9.1 If the request is for SVOA, add 1 mL of the SVOA spiking solution (sect. 5.6).

7.9.2 If the request is for SIM, add 1 mL of the SIM Spiking solution (sect. 5.7).

7.9.3 If the request is for SVOA/SIM, add 1 mL of the SVOA spiking solution and 1 mL of the SIM Spiking solution to appropriate LCS/LCSD and/or MS/MSD. (sect's 5.6 and 5.7).

7.9.4 If the request is for SVOA Appendix IX, add 1 mL of the SVOA Appendix IX spiking solution and 1 mL of the SVOA spiking solution (sect's 5.6 and 5.8).

7.10 Place each of the Soxhlet extractors in a heating mantle and lower the Allihn cooling water condensers into the 45/50 joints of the extractors. Save the pieces of aluminum foil for covering the Soxhlets when the extraction is complete. Switch on the individual heating mantles and be sure that the Rheostat of the variable transformer is set to 55-60% of the output voltage. Once the methylene chloride begins to boil and the Soxhlet begins to cycle (solvent will immerse the sample and collect in the Soxhlet until the level reaches that of the small siphon tube and then begin to spill over into the extract flask), re-check the apparatus' for leaks. Allow the samples to extract for 18-24 hours. Be sure the chiller/recirculator temperature is set low enough to provide enough cooling capacity for the number of extractions in the batch.

7.11 When the extraction is complete, allow the extracts to cool before dismantling. Tilt each extractor slightly to cause any remaining solvent in the sample chamber to drain through the siphon tube into the extract flask. This will help to cool the extract flask and make the apparatus easier to dismantle. Remove the Allihn condenser and replace the aluminum foil on top of the extractor. Move the extractors to a hood and detach the extractor from the extract flask. Try to drain as much solvent as possible from the extractor into the flask. **This is done by rinsing a glass tube in methylene chloride and pressing on the sample slightly so that as solvent as possible is drained into the extract flask.** Cover the flask with aluminum foil and

store in the interim extract storage refrigerator unless the extracts are to be concentrated the same day.

7.12 Immediately remove the extracted soil/sodium sulfate mixtures from the extractors using a square edge spatula, and dispose of in an appropriate solid waste container. It is important to do this soon after the extractors are dismantled, as the sample mixture will tend to "freeze" into a solid mass in the Soxhlet as the solvent dries.

CONCENTRATION OF EXTRACTS

7.13 Rinse the K-D glassware (flask, concentration tube, and Snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride before assembling. Add two boiling chips to the K-D. Insert 18.5 cm filter papers into short stem powder funnels and add ~ 2 inches of sodium sulfate crystals. Rinse the sodium sulfate in the assembled funnels with ~20-30 mLs of methylene chloride. Place the assembled K-D's under the funnels. Record the filter paper and sodium sulfate lot numbers in the extraction logbook.

7.14 Transfer the extracts to the K-D concentrator setups through the sodium sulfate in the funnels. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to pouring through the sodium sulfate. After pouring all of the extract volume through the sodium sulfate, rinse the extract flask three times with ~ 2 – 3 mLs of methylene chloride. Add the rinsings through the sodium sulfate to complete a quantitative transfer. Rinse the sodium sulfate with ~ 15 mLs of methylene chloride and allow draining.

7.15 All samples should go through GPC cleanup except if time does not permit. Refer to the current revision of Katahdin SOP CA-513, Extract Cleanup Using Gel Permeation Chromatography, for appropriate concentrating procedures.

7.16 If samples are not to be GPC'd, when time does not permit, follow Steps 7.17 through 7.22 to concentrate extracts to final volume of 1 mL.

7.17 Transfer the labels from the extract flasks to the K-Ds. Remove the funnel and attach a 3-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride.

7.18 Place the K-D in a hot water bath (75-85°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 4-6 mL, remove the K-D from the water bath. **Do not allow the evaporator to go dry. If the sample extract does go dry, re-**

extraction must occur immediately. Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of methylene chloride. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL methylene chloride.

7.19 Reduce the extract in the concentrator tube to approximately 1 mL using the nitrogen blow-down apparatus. The bath temperature must be no higher than the boiling point of the solvent (39°C for methylene chloride). Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with ≈1 mL of methylene chloride. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N_2 sparging pipet closer to the surface of the extract to expedite the concentration. Record the temperature of the water in the nitrogen evaporation water bath in the extraction logbook, also note any problems or extract losses, if they occur.

7.20 When the apparent volume reaches slightly less than 1 mL, remove the concentrator tube and allow it to cool.

7.21 Complete the quantitative transfer of the extract to a 1.8 mL vial by using methylene chloride. Adjust the volume of the methylene chloride extract to 1.0 mL using the 1.8 mL reference vial for volume comparison.

7.22 Label the vial with lab sample number, extraction date, matrix and analysis. Store extract vials at a temperature of 4 ± 2 °C until ready for analysis. Indicate in the extractions logbook the box number and “tray location” of the individual extract vials.

8 QUALITY CONTROL AND ACCEPTANCE CRITERIA

A method blank must be extracted for each and every item listed below:

- Each sample matrix (soil, water)
- Each day of extraction (24 hours midnight - midnight)
- Each extraction method or level
- Every 20 samples extracted in a 24-hour period

A laboratory control sample (LCS) is required for each and every item listed below:

- Each sample matrix
- Each extraction method or level
- Every extraction batch of twenty or fewer samples

Refer to the current revision of the applicable Katahdin SOP for analysis of extractable semivolatile organics for quality control acceptance criteria.

Each extractions analyst must demonstrate proficiency in performing the extractions that prepare samples for analysis. Demonstration consists of preparation (extraction), by the analyst, of at least four aliquots which are then analyzed according to the analytical method in question. These QC samples must meet all quality control acceptance limits. Demonstration must be documented by use of a form which summarizes the results of the analysis of these aliquots, calculated percent recoveries, and standard deviation. Demonstration of proficiency must be done one time per analyst initially and then annually thereafter. Refer to SOPs QA-805 and QA-807, current revision.

If, upon analysis of the extracted samples, it is discovered that quality control acceptance criteria have not been met, all associated samples must be evaluated against all the QC. In some cases data may be reported, perhaps with narration, while in other cases, other corrective action may be taken. The corrective actions may include re-extraction of the samples associated with the quality control sample that did not meet acceptance criteria, or may include making new reagents and standards if the standardization is suspect. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases “qualified” data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Organic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method

Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the applicable analytical SOP for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste-Physical/Chemical Methods, Method 3540C, SW-846, Third Edition, Updates I, II, IIA, IIB, and III Revised December 1996, US EPA.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.1, 04/22/09.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

LIST OF TABLES AND FIGURES

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

TABLE 1

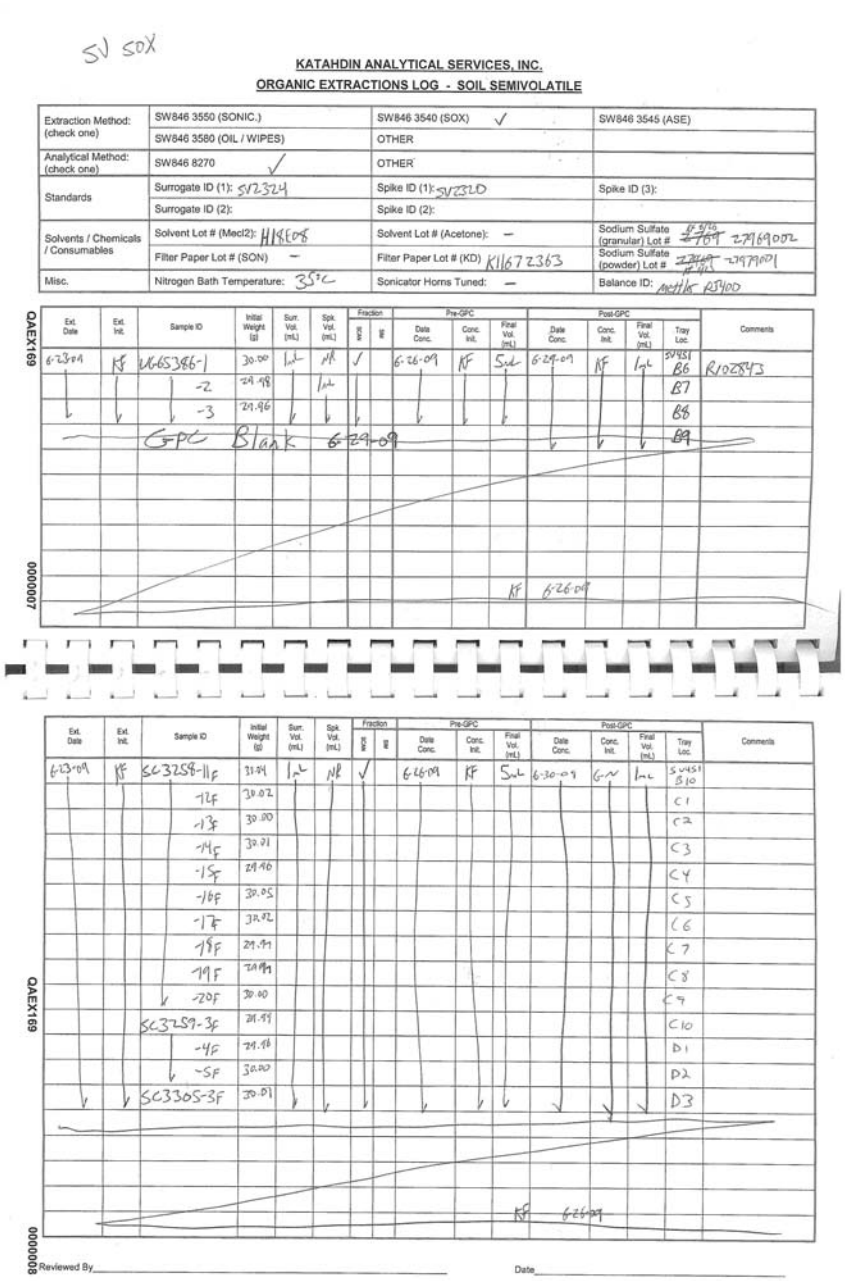
SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-526-06	METHOD 3540, current revision
Apparatus/Materials	1. short stem funnels	2. drying columns
Reagents		
Sample preservation/ handling		
Procedures	1. Use 30 grams of sample and 30 grams of sodium sulfate 2. Place a plug of glass wool in soxhlet then add sample 3. Use 250 mL of methylene chloride for extraction 4. Extract the sample for 18 - 24 hours 5. Extract dried using Na_2SO_4 in short stem funnels 6. Rinse the extract flask three times with ~ 2 – 3 mLs of methylene chloride then rinse the sodium sulfate with ~ 15 mLs of methylene chloride to complete a quantitative transfer 7. no apparatus height specification for concentration on water bath 8. Water bath at 75-85 deg C 9. Sample removed from water bath when volume reaches ~6 mL	1. Use 10 grams of sample and 10 grams of sodium sulfate. 2. Place sample between 2 plugs of glass wool 3. Use 300 mL of methylene chloride for extraction 4. Extract the sample for 16 - 24 hours at 4 - 6 cycles/hour 5. Extract dried using Na_2SO_4 in drying columns 6. Wash the extractor flask and sodium sulfate column with 100 to 125 mL of extraction solvent to complete the quantitative transfer 7. partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-20min 8. Water bath at 15-20 deg C above solvent boiling point 9. Sample removed from water bath when volume reaches 1-2 mL
QC - Spikes	1. Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1. Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - LCS	1. Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1. Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - Accuracy/Precision		
QC – MDL		

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

FIGURE 1

EXAMPLE OF LOGBOOK PAGE



SV SOX

KATAHDIN ANALYTICAL SERVICES, INC.
ORGANIC EXTRACTIONS LOG - SOIL SEMIVOLATILE

Extraction Method: (check one)	SW846 3550 (SONIC.)	SW846 3540 (SOX) ✓	SW846 3545 (ASE)
	SW846 3580 (OIL / WIPES)	OTHER	
Analytical Method: (check one)	SW846 8270 ✓	OTHER	
Standards	Surrogate ID (1): SV2324	Spike ID (1): SV23LD	Spike ID (3):
	Surrogate ID (2):	Spike ID (2):	
Solvents / Chemicals / Consumables	Solvent Lot # (MeCl2): HI86098	Solvent Lot # (Acetone): —	Sodium Sulfate (granular) Lot # 27169002
	Filter Paper Lot # (SON) —	Filter Paper Lot # (KD) K1167363	Sodium Sulfate (powder) Lot # 27969001
Misc.	Nitrogen Bath Temperature: 35°C	Sonicator Horns Tuned: —	Balance ID: AN1116 A5900

QAEX169 0000007

Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spk. Vol. (mL)	Fraction WSE	Fraction WE	Pre-GPC Date Conc.	Pre-GPC Conc. Init.	Pre-GPC Final Vol. (mL)	Post-GPC Date Conc.	Post-GPC Conc. Init.	Post-GPC Final Vol. (mL)	Tray Loc.	Comments
6-23-09	KF	LL65386-1	30.00	1mL	NA	✓		6-26-09	KF	5mL	6-29-09	KF	1mL	SV451 B6	R102843
		-2	29.98	1mL										B7	
		-3	29.96	1mL										B8	
		GPC Blank	6-29-09											B9	
										KF	6-26-09				

Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spk. Vol. (mL)	Fraction WSE	Fraction WE	Pre-GPC Date Conc.	Pre-GPC Conc. Init.	Pre-GPC Final Vol. (mL)	Post-GPC Date Conc.	Post-GPC Conc. Init.	Post-GPC Final Vol. (mL)	Tray Loc.	Comments
6-23-09	KF	SC3258-11F	31.04	1mL	NA	✓		6-26-09	KF	5mL	6-30-09	GN	1mL	SV451 B10	
		-12F	30.02											C1	
		-13F	30.00											C2	
		-14F	30.21											C3	
		-15F	29.90											C4	
		-16F	30.05											C5	
		-17F	30.92											C6	
		18F	29.71											C7	
		-19F	29.94											C8	
		-20F	30.00											C9	
		SC3259-3F	29.99											C10	
		-4F	29.96											D1	
		-5F	30.00											D2	
		SC3305-3F	30.01											D3	
										KF	6-26-09				

QAEX169 0000008

Reviewed By_____ Date_____

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

FIGURE 2

LCS/MATRIX SPIKE COMPONENT LIST

BASE/NEUTRALS	
1-Methylnaphthalene	Bis (2-chloroethoxy) methane
1,1-Biphenyl	Bis (2-chloroethyl) ether
1,2,4-Trichlorobenzene	Bis (2-Chloroisopropyl) ether)
1,2-Dichlorobenzene	Bis (2-ethylhexyl) adipate
1,3-Dichlorobenzene	Bis (2-ethylhexyl) phthalate
1,4-Dichlorobenzene	Butylbenzyl phthalate
1,4-Dioxane	Caprolactam
2,4-Dinitrotoluene	Carbazole
2,6-Dinitrotoluene	Chrysene
2-Chloronaphthalene	Dibenz (a, h) anthracene
2-Methylnaphthalene	Dibenzofuran
2-Nitroaniline	Diethyl adipate
3,3'-Dichlorobenzidine	Diethyl phthalate
3-Nitroaniline	Dimethyl phthalate
4-Bromophenylphenyl ether	Di-n-butylphthalate
4-Chloroaniline	Di-n-octyl phthalate
4-Chlorophenylphenyl ether	Fluoranthene
4-Nitroaniline	Fluorene
Acenaphthene	Hexachlorobenzene
Acenaphthylene	Hexachlorobutadiene
Acetophenone	Hexachlorocyclopentadiene
Aniline	Hexachloroethane
Anthracene	Indeno (1,2,3-cd) pyrene
Atrazine	Isophorone
Azobenzene	Naphthalene
Benzaldehyde	Nitrobenzene
Benzidine	N-Nitrosodimethylamine
Benzo (a) Anthracene	N-Nitroso-di-n-propylamine
Benzo (a) pyrene	N-Nitrosodiphenylamine
Benzo (b) fluoranthene	Phenanthrene
Benzo (ghi) perylene	p-toluidine
Benzo (k) fluoranthene	Pyrene
Benzyl alcohol	Pyridine

ACIDS		
2, 3, 4, 6-Tetrachlorophenol	2-Chlorophenol	Benzoic acid
2,4,5-Trichlorophenol	2-Methylphenol	Ethyl methanesulfonate
2,4,6-Trichlorophenol	2-Nitrophenol	Methyl methanesulfonate
2,4-Dichlorophenol	4,6-Dinitro-2-methylphenol	Pentachlorophenol
2,4-Dimethylphenol	4-Chloro-3-methylphenol	Phenol
2,4-Dinitrophenol	4-Methylphenol	
2,6-Dichlorophenol	4-Nitrophenol	

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

FIGURE 3

APPENDIX IX LCS/MATRIX SPIKE COMPONENT LIST

1,2,4,5-Tetrachlorobenzene	Hexachloropropene
1,3,5-Trinitrobenzene	Isodrin
1,4-Naphthoquinone	Isosafrole
1-Chloronaphthalene	Kepone
1-Naphthylamine	m-Dinitrobenzene
2,4-D	Methapyrilene
2-Acetyl aminofluorene	Methyl parathion
2-Naphthylamine	n-Nitrosodiethylamine
2-Picoline	n-Nitrosodi-n-butylamine
3,3-Dimethylbenzidine	n-Nitrosomethylethylamine
3-Methylcholanthrene	n-Nitrosomorpholine
4-Aminobiphenyl	n-Nitrosopyrrolidine
4-Nitroquinoline-1-oxide	n-Nitrotrosopiperidine
5-Nitro-o-toluidine	O,O,O-Triethyl phosphorothioate
7,12-Dimethylbenz(a)anthracene	o-Toluidine
a,a-Dimethylphenethylamine	Parathion
Acetophenone	p-Dimethylaminoazobenzene
Aramite	Pentachlorobenzene
Chlorobenzilate	Pentachloronitriobenzene
Diallate	Phenacetin
Dibenz(a,j)acridine	Phorate
Dimethoate	p-Phenylenediamine
Dinoseb	Pronamide
Diphenylamine	Safrole
Disulfoton	Silvex (2,4,5-TP)
Famphur	Sulfotep
Hexachlorophene	Thionazin

TITLE: PREPARATION OF SEDIMENT/SOIL AND TISSUE SAMPLES BY ACCELERATED SOLVENT EXTRACTION USING METHOD 3545 FOR SUBSEQUENT EXTRACTABLE PESTICIDE and PCB ANALYSIS

Prepared By: Galen Nickerson Date: 6-23-06

Approved By:

Department Manager: Peter Lee Date: 6-23-06

Operations Manager: Deborah J. Nadeau Date: 6.23.06

QA Officer: Leslie Dimond Date: 6-23-06

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Sect. 1.4 - Added solvent and acid waste streams. Changed solvent in sect. 7.4.2 from hexane to CH_2Cl_2. Updated logbook example. Added recording of solvent, Na_2SO_4, hydromix, filterpaper Lot #'s as well as the temperature of the N_2 waterbath in the logbook.	LAD	07/08	07/08
02	Updated section 4 with current materials. Updated section 7 with current techniques. Changed when Hexane is added during the solvent exchange process. Changed all weights to record to 0.05g. Updated logbook example. Added CA-108 reference for subsampling.	LAD	08/09	08/09

TITLE: **PREPARATION OF SEDIMENT/SOIL AND TISSUE SAMPLES BY ACCELERATED SOLVENT EXTRACTION USING METHOD 3545 FOR SUBSEQUENT EXTRACTABLE PESTICIDE and PCB ANALYSIS**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy _____ of document **SOP CA-537-02,** titled **Preparation of Sediment/Soil and Tissue Samples by Accelerated Solvent Extraction Using Method 3545 for Subsequent Extractable Pesticide and PCB Analysis**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy _____ of document **SOP CA-537-02,** titled **Preparation of Sediment/Soil and Tissue Samples by Accelerated Solvent Extraction Using Method 3545 for Subsequent Extractable Pesticide and PCB Analysis**.

Recipient: _____ Date: _____

TITLE: **PREPARATION OF SEDIMENT/SOIL AND TISSUE SAMPLES BY ACCELERATED SOLVENT EXTRACTION USING METHOD 3545 FOR SUBSEQUENT EXTRACTABLE PESTICIDE and PCB ANALYSIS**

1.0 SCOPE AND APPLICATION

Method 3545 is a procedure for extracting water insoluble or slightly water soluble semivolatile organic compounds from soils, clays, sediments, sludges, waste solids and tissues samples. This Pressurized Fluid Extraction (PFE) method uses elevated temperature (100°C or 175°C) and pressure (1500 - 2000 psi) to achieve analyte recoveries equivalent to those from Soxhlet extraction, using less solvent and taking significantly less time than the Soxhlet procedure.

This method is applicable to the extraction of organochlorine pesticides and PCBs. Organochlorine pesticides and PCBs may then be analyzed by a variety of chromatographic procedures.

1.1 Definitions

ANALYTICAL BATCH: 20 or fewer samples which are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods.

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

TITLE: **PREPARATION OF SEDIMENT/SOIL AND TISSUE SAMPLES BY ACCELERATED SOLVENT EXTRACTION USING METHOD 3545 FOR SUBSEQUENT EXTRACTABLE PESTICIDE and PCB ANALYSIS**

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the extraction of samples for pesticide and PCB analysis. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training".

It is the responsibility of all Katahdin technical personnel involved in the extraction of samples for pesticide and PCB analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that the members of his/her group follow this SOP, to assure that their work is properly documented, and to indicate periodic review of the pertinent logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves, and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their department manager, or designee, appropriate for the job functions they will perform.

1.4 Waste Disposal

Wastes generated during the preparation of samples must be disposed of in accordance with the procedures described in the current revision of the Katahdin Hazardous Waste Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the rinsing of glassware etc. should be disposed of in the "D" waste stream satellite accumulation area nearest the point of generation. Acetone and hexane are considered flammable waste, and should be disposed of in the "O" waste stream satellite accumulation area nearest the point of generation. Post-extraction soil samples, used glass wool, and sodium sulfate waste should be disposed of in the soil with organics "I" waste stream satellite accumulation area nearest the point of generation. Acid waste generated during the cleanup of PCB samples should be disposed of in the "K" satellite accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

2.0 SUMMARY OF METHOD

Samples are prepared for extraction by weighing out a specific quantity of sample and mixing said quantity with pelletized diatomaceous earth (Hydromatrix) to remove moisture. The sample is then loaded into an extraction cell and placed on the Accelerated Solvent Extractor (ASE).

The extraction cell containing the sample is heated to the analyte-specific extraction temperature (see section 7.0), pressurized with hexane and extracted for a period of time outlined in section 7.0.

The solvent is collected from the extraction cells into 60 mL vials and allowed to cool. The solvent is then concentrated and, as needed, exchanged into solvent compatible with the cleanup or determinative step being employed.

3.0 INTERFERENCES

Contaminants in solvents, reagents, glassware, and other sample processing hardware may cause method interferences such as discrete artifacts and/or elevated baselines in the total ion current profiles (TICPs). All of these materials routinely must be demonstrated to be free from interferences under the conditions of the analysis by running laboratory method blanks. Interferences caused by phthalate esters can pose a major problem in semivolatile organics analysis because many phthalates are also target analytes. Common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, so cross-

contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory. At no time may gloves that have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Additionally, whenever possible plastic items in this lab must be replaced with metal or Teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure clean glassware and apparatus are used, pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis. Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to interferences, further cleanup of the sample extract may be needed to minimize interferences.

4.0 APPARATUS AND MATERIALS

4.1. Pressurized fluid extraction device: Dionex Accelerated Solvent Extractor, Model number ASE 200 with appropriately sized extraction cells. The cells will accommodate 15 g of sample and are made of stainless steel which is capable of withstanding the pressure requirements necessary for this procedure.

4.2. Analytical Balance: Mettler PJ4000 balance capable of weighing to 0.01 g

4.3. Vials for the collection of extracts: Dionex 60 ml pre-cleaned, open to screw cap with PTFE-lined silicone septum.

4.4. Filter disk for extraction vessels: Dionex 1.91 cm, Type D28.

4.5. 400 ml beakers

4.6 Ottawa Sand (Fisher P/N 523-3)

4.7 Kuderna-Danish (K-D) apparatus

4.7.1 Concentrator tube - 10-mL

4.7.2 Evaporation flask - 500-mL

4.7.3 Snyder column - Three-ball macro

4.8 Nitrogen evaporation (N-EVAP) apparatus.

4.9 Stainless steel spatula.

4.10 Porcelain mortar and pestle.

4.11 Boiling stones, 12 mesh silicon carbide (carborundum) – pre-purified by Soxhlet extraction in methylene chloride.

4.12 Vials - Glass, 12 mL and 4 mL capacity

4.13 500 ul syringes

4.14 Funnel powder, glass.

4.15 Filter paper, 18.5 cm, Fisherbrand or Whatman 114V (or equivalent)

4.16 Water bath or steam bath capable of maintaining a temperature of at least 85°C.

5.0 REAGENTS

5.1 Aluminum oxide (Alumina, acid), Brockman activity I, 60-325 mesh

5.2 Drying agents

5.2.1 Pelletized diatomaceous earth (Hydromatrix) - Fisher P/N.

5.2.2 Sodium sulfate crystals, Na_2SO_4.

5.3 Extraction solvents

5.3.2 Methylene Chloride - Pesticide grade or equivalent. Lot must be verified by concentrating ≈ 300 mL to 1 mL and evaluating by both GC/MS and GC/FID analyses.

5.3.3 Hexane - Pesticide grade or equivalent. Lot must be verified by concentrating 200-300 mLs to 1 mL followed by GC/ECD analysis.

5.3.4 Acetone – Pesticide grade or equivalent. Lot must be verified by concentrating ≈ 300 mL to 1 mL followed by GC/MS, GC/ECD, and GC/FID analyses.

5.3.5 Organochlorine pesticides and PCBs may be extracted with acetone/methylene chloride (1:1,v/v) or hexane (100%).

5.3.6 Other solvent systems may be employed, provided that the analyst can demonstrate adequate performance for the analytes of interest in the sample matrix

CAUTION: For best results with very wet samples (e.g., ≥ 30% moisture), reduce or eliminate the quantity of hydrophilic solvent used.

5.4 Pre-purified nitrogen is used to purge and/or pressurize the extraction cell.

5.5 Spiking Solutions

5.5.1 Pesticide/PCB surrogate spiking solution - Prepare a solution of decachlorbiphenyl (DCB) and tetrachloro-meta-xylene (TCMX) at a concentration of 1 ug/mL in acetone. Store the solution at –10 to -20 °in a Teflon sealed container. Solution must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.

5.5.2 Pesticide/PCB matrix spike/Lab control sample spiking solution - Prepare separate spiking solutions for Pesticides and one for PCBs in pesticide grade methanol and acetone, respectively, that contain all target analytes listed below:

Pesticide Spiking Solution

Analyte	ug/mL	Analyte	ug/mL
4,4'-DDD	0.5	Endosulfan I	0.5
4,4'-DDE	0.5	Endosulfan II	0.5
4,4'-DDT	0.5	Endosulfan Sulfate	0.5
Aldrin	0.5	Endrin	0.5
alpha-BHC	0.5	Endrin Aldehyde	0.5
beta-BHC	0.5	Endrin Ketone	0.5
delta-BHC	0.5	gamma-BHC (Lindane)	0.5
Dieldrin	0.5	Heptachlor	0.5
alpha-Chlordane	0.5	Heptachlor Epoxide	0.5
gamma-Chlordane	0.5	Methoxychlor	0.5

PCB Spiking Solution

Analyte	ug/mL
Aroclors 1016/1260	5.0

Store the solution at –10 to -20 °C in a Teflon sealed container. The solution must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

TITLE: **PREPARATION OF SEDIMENT/SOIL AND TISSUE SAMPLES BY ACCELERATED SOLVENT EXTRACTION USING METHOD 3545 FOR SUBSEQUENT EXTRACTABLE PESTICIDE and PCB ANALYSIS**

Holding time for extraction of sediment/soil and tissue samples for Method 3545 is 14 days from date of sample collection, although the analyst should be aware that actual holding times employed may be project/program specific.

Store all extracts at 4°C (± 2°C) in the dark in labeled Teflon-sealed containers. See SOP SD-902, "Sample Receipt and Internal Control," current revision, for storage areas and temperature maintenance procedures.

7.0 PROCEDURES

7.1 Preparing the Accelerated Solvent Extractor

7.1.1 Before extractions can begin, the system must first go through its daily maintenance checks. To begin, turn on the system and nitrogen gas tank. Check the system pressure to ensure that the solvent pressure is at 10 psi, air is at 50 psi, and compression is at 130 psi. Next, check the solvent bottle and fill with the appropriate solvent if necessary. At this time the rinse and waste vials should be emptied and four 60 ml vials need to be placed in the rinse positions of the sample collection carousel. Record the solvent lot number in the extraction logbook.

7.1.2 The system must now be rinsed before the analysis can begin. Press the rinse button on the touch pad. The system will immediately begin its rinse cycle. This rinse procedure should be repeated at least three times before any samples are extracted to ensure that all contaminants are removed from the system.

7.1.3 To prepare extraction cells, disassemble the ASE extractor cells and clean each piece with soapy water. Next rinse each piece with acetone to ensure that all water is removed. Next, rinse the body and two screw caps three times with methylene chloride. Be sure you are using a solvent whose lot has been checked. Inspect each screw cap to ensure that the o-rings are not crushed and in need of replacement. O-rings should be replaced after approximately 50 extractions to ensure proper fit. Cell bodies should also be checked for nicks and dings at each end. If any dings are present the cell should be placed out of rotation until it can be repaired. If any instrument maintenance is necessary record the maintenance performed in the instrument maintenance logbook.

7.1.4 Once the extraction cells have been rinsed replace the bottom cell screw cap. The bottom of the cell body is the end that is imprinted with Dionex.

Next, place 2 19.8 mm filter disks into the top of the cell body and push it to the bottom of the cell with the plunger that was supplied with the system. Record the lot numbers of the filter disks in the extraction logbook.

If extracting pesticides/PCBs from tissue samples weigh out 5.00 ±1.00 g of aluminum oxide (alumina, acid) into cell and place a second filter on the top of alumina. Avoiding matrix affect interference for tissue sample, the aluminum oxide is used to remove lipid. Record the lot numbers of the aluminum oxide and filters in the extraction logbook.

7.1.5 Label each extraction cell and clean, rinsed, 60 ml collection vial with the appropriate sample ID with a black marker.

7.2 Sample preparation

7.2.1 Sediment/soil samples - Decant and discard any water layer on a sediment sample. Mix the sample thoroughly with the stainless steel spatula. If the sample container is full to the extent that stirring the sample is impractical, try to remove the “best representative” aliquot from the jar based on color, particle size, moisture, etc. Discard any foreign objects such as sticks, leaves, and rocks.

7.2.2 Gummy, fibrous, or oily materials not amenable to grinding should be cut, shredded, or otherwise reduced in size to allow mixing and maximum exposure of the sample surfaces for the extraction. Materials such as glass, rubber, metal, etc. may not require mixing with Hydromatrix to disperse the sample. Plastic materials must be tested for degradation (melting) in methylene chloride prior to ASE extraction.

7.2.3 Tissue samples are blended prior to handling in extraction laboratory.

7.2.4 Refer to Katahdin Analytical Services SOP CA-108, current revision, “Basic Laboratory Technique” for more information regarding subsampling.

7.3 Sample Handling

The following steps should be performed rapidly to avoid loss of the more volatile extractables.

7.3.1 Weigh out 15.00 ± 0.05g portion of sample into a 400 ml beaker. For tissue sample, weigh out a 5.00 ± 0.05g portion of sample into a 400 ml beaker.

Record sample weight to the nearest 0.05g in the appropriate extraction logbook.

7.3.2 Add enough Hydromatrix to the sample to produce a "free-flowing" mixture, around 5g. For tissue sample, pulverize the mixture (sample-hydromatrix) using the porcelain mortar-pestle apparatus. At this point don't add an excessive amount because more Hydromatrix can be added later in the procedure. This ensures that the sample will properly fit in the extraction cell. The amount of Hydromatrix will depend upon the moisture content of the sample (e.g., low moisture content will require less Hydromatrix). Mix well with a spatula. Keep the spatula in the sample beaker and cover the beaker with aluminum foil. Record the Hydromatrix lot number in the extraction logbook.

7.3.3 A method blank must be prepared with each extraction batch, not to exceed 20 client samples. To prepare a method blank, weigh out a 5.00 ± 0. 05g portion of Ottawa sand for tissue sample batch or 15.00 ± 0.05g portion for sediment/soil sample batch into a 400 ml beaker. Record the weight to the nearest 0.05 g in the appropriate extraction logbook. Add enough Hydromatrix to the sand to produce a "free-flowing" mixture. At this point don't add an excessive amount because more Hydromatrix can be added later in the procedure. This ensures that the sample will properly fit in the extraction cell. Mix well with a spatula. Keep the spatula in the sample beaker and cover the beaker with aluminum foil.

7.3.4 A laboratory control sample (LCS) must be prepared with each extraction batch, not to exceed 20 client samples. If no MS/MSD is to be prepared a laboratory control sample duplicate must be prepared. To prepare an LCS (and LCSD), weigh out a 15.00 ± 0.05g portion of Ottawa sand into a 400 ml beaker; for tissue sample, weigh out a 5.00 ± 0. 05 g of Ottawa sand into a 400 mL beaker. Record the weight to the nearest 0. 05 g in the appropriate extraction logbook. Add enough Hydromatrix, approximately 5g to the sand to produce a "free-flowing" mixture. At this point don't add an excessive amount because more Hydromatrix can be added later in the procedure. This ensures that the sample will properly fit in the extraction cell. Mix well with a spatula. Keep the spatula in the sample beaker and cover the beaker with aluminum foil. **If extracting both pesticide and PCB separate LCS/D and MS/D need to be prepared.**

7.3.5 A matrix spike/matrix spike duplicate (MS/MSD) set must be prepared for every 20 samples. To prepare MS/MSD, weigh out a 15.00 ± 0.05g portion of designee sample into a 400 ml beaker. For tissue sample, weigh out a 5.0 ± 0.05g portion of designee sample into a 400 mL beaker. Record the

weight to the nearest 0.05g in the appropriate extraction logbook. Add enough Hydromatrix approximately 5g to the sand to produce a "free-flowing" mixture. At this point don't add an excessive amount because more Hydromatrix can be added later in the procedure. This ensures that the sample will properly fit in the extraction cell. Mix well with a spatula. Keep the spatula in the sample beaker and cover the beaker with aluminum foil.

7.3.6 Once all the QC and field samples have been weighed and mixed with a portion of Hydromatrix, find the appropriately labeled extraction cell and place the cell funnel, rinsed three times in methylene chloride, on the open end. This funnel was supplied with the system. Pour the contents of the beaker into the extraction cell. Carefully scrape all the mixtures from the beaker walls so that no more than 1% remains behind in the beaker. If there is head space remaining in the extraction cell fill the remaining space with more Hydromatrix by pouring the sample back into the beaker and mixing the new Hydromatrix into the sample. This will minimize the amount of solvent used. Again, pour the sample back into the same extraction cell and cover with a screw cap. Rinse the cell funnel with extraction solvent between each sample.

7.3.7 To all tissue samples, method blank, LCS, and MS/MSD add 0.2 mL of Pest/PCB Surrogate spiking solution using a pre-rinsed 500 uL gas tight syringe. To all sediment/soil samples and QC being extracted add 0.5 mL of Pest/PCB surrogate spiking solution using a pre-rinsed 0.5 mL gas-tight syringe. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent when spiking is complete. To LCS and MS add 0.5 mL of appropriate spike solution to each.

7.3.8 Replace all screw caps and ensure that each is properly tightened. Place each extraction cell into the ASE sample carousel. Place the corresponding collection vial into the appropriate location on the sample collection carousel. Be certain that the cell and vial are in the same number position of the carousel. In order to avoid a possible carryover, samples should be placed in order of their cleanness.

7.3.9 Press the menu button on the ASE touch pad. Choose option number one and press enter. Select method number one for Pesticide and PCB analysis, press enter. Press start on the
touch pad. The extraction process will begin and takes approximately 14 minutes per sample to complete.

NOTE: Recommended extraction conditions:

For Pest/PCB (saved in the instrument as method one)
Oven temperature: 100°C
Pressure: 1500 - 2000 psi
Static time: 5 min (after 5 min pre-heat equilibration)
Flush volume: 60% of the cell volume
Nitrogen purge: 90 sec at 150 psi (purge time may be extended for larger cells)
Static Cycles: 1

Optimize the conditions, as needed, according to the manufacturer's instructions. In general, the pressure is not a critical parameter, as the purpose of pressurizing the extraction cell is to prevent the solvent from boiling at the extraction temperature and to ensure that the solvent remains in intimate contact with the sample. Any pressure in the range of 1500 - 2000 psi should suffice.

Once established, the same pressure should be used for all samples extracted for the same analysis type.

As stated above, the recommended conditions have been saved as methods on the instrument. If it becomes necessary to re-program conditions, follow the above guidelines.

7.3.10 Once the extraction process is complete and the instrument is idle remove the sample extracts and label with appropriate sticker. They are now ready to be stored in the interim extract storage refrigerator unless the samples are to be concentrated the same day.

7.3.11 Remove the extraction cells from the ASE and empty their contents into the appropriate solid waste container.

7.3.12 The extract is now ready for concentration, cleanup, or analysis, depending on the extent of interferents and the determinative method to be employed. Certain cleanup and/or determinative methods may require a solvent exchange prior to cleanup and/or sample analysis.

7.4 Concentration of Extracts

7.4.1 If samples are to be GPC'd, refer to the current revision of Katahdin SOP CA-513, Extract Cleanup Using Gel Permeation Chromatography, for appropriate concentrating procedures.

7.4.2 If samples are not to be GPC'd follow steps 7.4.3 through 7.4.9 to concentrate extracts to final volume of 5.0 mLs for soil/sediment samples and 2.0 mL for tissue samples

7.4.3 Before assembling, rinse the K-D glassware (flask, concentration tube, and Snyder column, including the ground glass joints on the flask and columns) three times with hexane if using 100 % hexane for Pesticide/PCB extraction, if not rinse three times with methylene chloride. Add two boiling chips to the K-D. Insert 18.5 cm filter papers into short stem powder funnels and add ≈ 2 inches of sodium sulfate crystals. Rinse the sodium sulfate in the assembled funnels with ≈20-30 mLs of methylene chloride. Place the assembled K-D's under the funnels. Visually examine each 60mL vial of sample. If there is excessive moisture in the sample, biphasic layering will be seen. If this occurs, add approximately 1 inch of sodium sulfate crystals to the vial. This should remove most of the moisture. Record the lot number of the sodium sulfate in the extraction logbook.

7.4.4 For a solvent exchange, no GPC, add approximately 50 mL Hexane to funnel and let drain through. Since methylene chloride has a lower boiling point than Hexane, this will result in a final extract in hexane only. Record the lot number of the solvent in the extraction logbook.

7.4.5 Transfer the extracts to the K-D concentrator setups through the sodium sulfate in the funnels. This is the drying step, which is required to remove residual water from the extracts. After pouring all of the extract volume through the sodium sulfate, rinse the extract vial three times with methylene chloride. Add the rinsings through the sodium sulfate to complete a quantitative transfer. Rinse the sodium sulfate with ≈ 15 mLs of methylene chloride and allow draining.

7.4.6 Transfer the labels from the extract flasks to the K-Ds. Remove the funnel, add one or two clean boiling stones to the K-D evaporative flask and attach a 3-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of hexane.

7.4.7 Place the K-D in a hot water bath (85-90°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 6 mL, remove the K-D from the water bath. Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of hexane. Remove the Snyder column. Wipe off

any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL hexane.

7.4.8 Reduce the extract in the concentrator tube to approximately 1 mL using the nitrogen blow-down apparatus. The bath temperature must be no higher than the boiling point of the solvent (39°C for methylene chloride). Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with ≈1 mL of hexane. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N_2 sparging pipet closer to the surface of the extract to expedite the concentration. Record the temperature of the water in the nitrogen evaporation water bath in the extraction logbook, also note any problems or extract losses, if they occur.

7.4.9 Complete quantitative transfer of the extract to a vial by using hexane. Adjust the volume of the hexane extract to 5 mL in a 12mL vial using the appropriate “reference vial” for volume comparison. For Pesticides/PCB from tissue samples, adjust the volume of hexane extract to 2.0 mL in a 4.0 mL vial using the appropriate “reference vial” for volume comparison.

7.4.10 Label the vial with lab sample number, extraction date, matrix and analysis. Store extract vials at a temperature of 4 ± 2 °C until ready for analysis. Indicate in the extractions logbook the box number and “tray location” of the individual extract vials.

7.4.11 All sample extracts for 8082 PCB analysis must undergo a sulfuric acid wash (cleanup) prior to analysis. Sample extracts for 8081 pesticide do not undergo further cleanup unless requested by the client. All soil/sediment sample extracts for combined 8081/8082 analyses must be split. One portion must be acid cleaned for 8082 analysis. The associated method blank must be split and acid-cleaned in the same fashion. PCB LCSs and matrix spikes are acid cleaned also. Pesticide LCSs and matrix spike samples are not subjected to further cleanup. Please refer to Katahdin SOP CA525 (current revision), Extract Cleanup Using Sulfuric Acid, for further instructions.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

A method blank must be extracted for each and every item listed below:

- Each sample matrix (soil, water)
- Each day of extraction (24 hours midnight - midnight)
- Each extraction method or level
- Every 20 samples extracted in a 24-hour period

A laboratory control sample (LCS) is required for each and every item listed below:

- Each sample matrix
- Each extraction method or level
- Every extraction batch of twenty or fewer samples

Refer to the current revision of the applicable Katahdin SOP for analysis of Pesticides and PCBs for quality control acceptance criteria.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Organic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the applicable analytical SOP for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste-Physical/Chemical Methods, Method 3545A, SW-846, Third Edition, Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, Revised February 2007, US EPA.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.1, 04/22/09.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

TITLE: **PREPARATION OF SEDIMENT/SOIL AND TISSUE SAMPLES BY ACCELERATED SOLVENT EXTRACTION USING METHOD 3545 FOR SUBSEQUENT EXTRACTABLE PESTICIDE and PCB ANALYSIS**

LIST OF TABLES AND FIGURES

TITLE: **PREPARATION OF SEDIMENT/SOIL AND TISSUE SAMPLES BY ACCELERATED SOLVENT EXTRACTION USING METHOD 3545 FOR SUBSEQUENT EXTRACTABLE PESTICIDE and PCB ANALYSIS**

TABLE 1

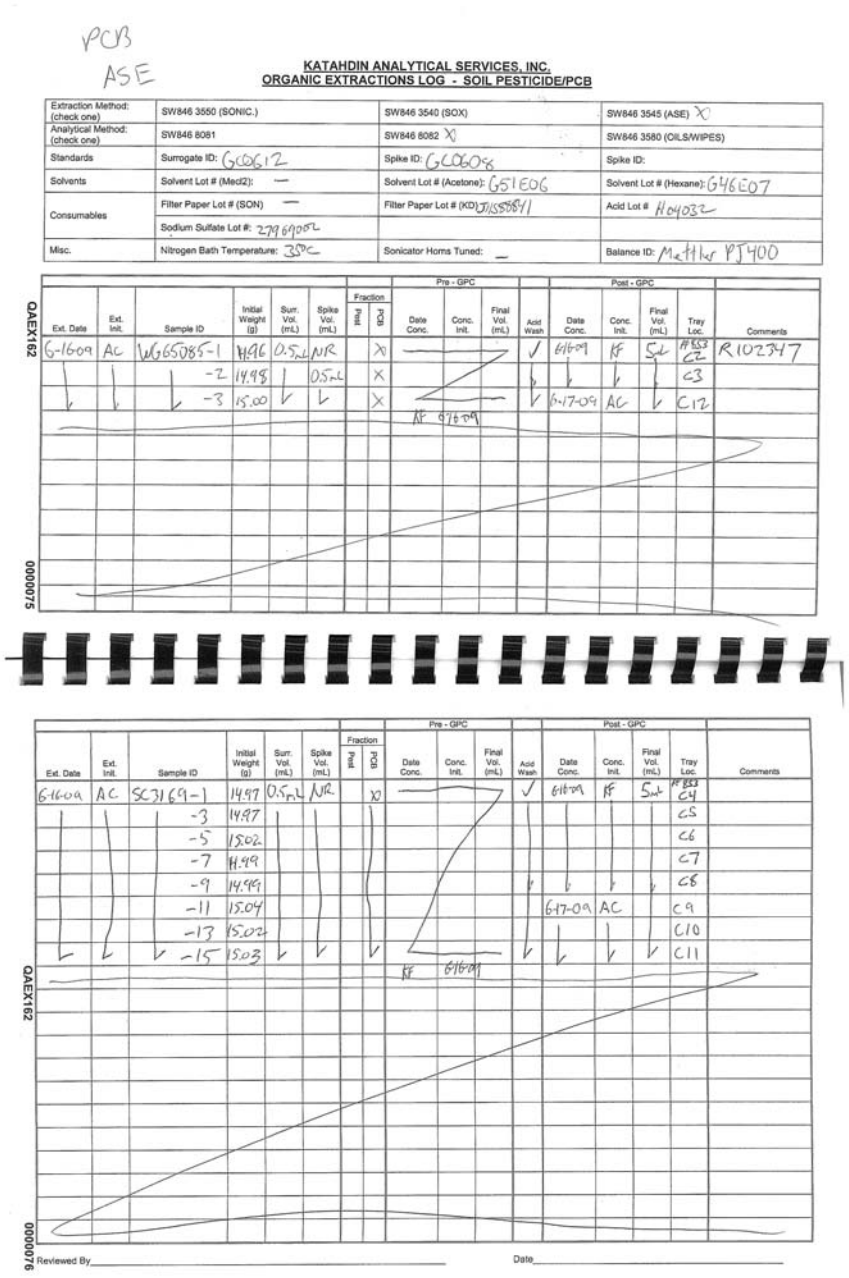
SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-537-02	METHOD 3545, current revision
Apparatus/Materials		
Reagents	Katahdin receives a certificate with each lot indicating sodium sulfate crystals were dried by the manufacturer at prescribed conditions.	Section 5.3.3 Drying agents should be purified by heating at 400 °C for 4 hours…
Sample preservation/ handling	Samples are not ground prior to mixing with the drying agent.	Section 7.3 Grind a sufficient weight of the dried sample…
Procedures	Section 7.3.9 Nitrogen purge for Pesticide/PCB and DRO: 90 sec at 150 psi Oven temperature for DRO: 175°C	Section 7.8 Recommended extraction conditions for all extractions: Nitrogen purge: 60 sec at 150 psi. Oven temperature: 100 °C
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

TITLE: **PREPARATION OF SEDIMENT/SOIL AND TISSUE SAMPLES BY ACCELERATED SOLVENT EXTRACTION USING METHOD 3545 FOR SUBSEQUENT EXTRACTABLE PESTICIDE and PCB ANALYSIS**

FIGURE 1

EXAMPLE OF LOGBOOK PAGE



PCB
ASE

KATAHDIN ANALYTICAL SERVICES, INC.
ORGANIC EXTRACTIONS LOG - SOIL PESTICIDE/PCB

Extraction Method: (check one)	SW846 3550 (SONIC.)	SW846 3540 (SOX)	SW846 3545 (ASE) X
Analytical Method: (check one)	SW846 8081	SW846 8082 X	SW846 3580 (OILS/WIPES)
Standards	Surrogate ID: GC0612	Spike ID: GC0605	Spike ID:
Solvents	Solvent Lot # (Med2): —	Solvent Lot # (Acetone): G51E06	Solvent Lot # (Hexane): G46E07
Consumables	Filter Paper Lot # (SON) —	Filter Paper Lot # (KD) JI1558/1	Acid Lot # H04032
	Sodium Sulfate Lot #: 279690SL		
Misc.	Nitrogen Bath Temperature: 35°C	Sonicator Horns Tuned: —	Balance ID: Mettler PJ400

Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spike Vol. (mL)	Fraction Prel	Fraction PCB	Pre-GPC Date Conc.	Pre-GPC Conc. Init.	Pre-GPC Final Vol. (mL)	Acid Wash	Post-GPC Date Conc.	Post-GPC Conc. Init.	Post-GPC Final Vol. (mL)	Tray Loc.	Comments
6-16-09	AC	WG65085-1	14.96	0.5mL	NR		X				✓	6/16/09	KF	5mL	#553 C2	R102347
		-2	14.98		0.5mL		X								C3	
		-3	15.00				X					6-17-09	AC		C12	
								KF 6/16/09								

QAEX162 0000075

Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spike Vol. (mL)	Fraction Prel	Fraction PCB	Pre-GPC Date Conc.	Pre-GPC Conc. Init.	Pre-GPC Final Vol. (mL)	Acid Wash	Post-GPC Date Conc.	Post-GPC Conc. Init.	Post-GPC Final Vol. (mL)	Tray Loc.	Comments
6-16-09	AC	SC31691-1	14.97	0.5mL	NR		X				✓	6/16/09	KF	5mL	#553 C4	
		-3	14.97												C5	
		-5	15.02												C6	
		-7	14.99												C7	
		-9	14.99												C8	
		-11	15.04									6/17/09	AC		C9	
		-13	15.02												C10	
		-15	15.03												C11	
								KF 6/16/09								

QAEX162 0000076

Reviewed By_____ Date_____

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

Prepared By: George Brewer Date: 11/97

Approved By:

Group Supervisor: George Brewer Date: 01/19/01

Operations Manager: John C. Buxton Date: 1/22/01

QA Officer: Deborah J. Nadeau Date: 1·22·01

General Manager: Dennis F. Keefe Date: 1/22/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 3010A	Format changes, added pollution prevention, block digester; revised database references; revised and added tables.	DN	1·22·01	1/22/01
02 3010A	Added wording allowing use of digestates for ICP-MS analysis. Added use of block digester as primary heating source & adjusted volumes. Revised standard solution names & concs. in Figures 3 & 4.	DN	8·29·02	8·29·02
03	Added Uranium to spiking solutions for LCS & MS/D. Removed the Internal Custody Record for Metals Digestates figure and reference.	LAD	04/06	04/06
04	Minor changes to Section 7 to reflect current practices. Updated Figure 1 - Sample Prep Logbook. Updated Figure 2 and 3 - Spike amounts.	LAD	05/09	05/09
05	Added references. Updated Figure 2 and 3 with correct spike information. Added CA-108 reference for subsampling information.	LAD	04/10	04/10

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ____ of document **SOP CA-604-05**, titled **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ____ of document **SOP CA-604-05**, titled **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**.

Recipient: _____ Date: _____

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedure utilized by Katahdin Analytical Services, Inc. personnel to solubilize metals in aqueous samples, wastes that contain suspended solids, and mobility-procedure extracts prior to analysis by inductively coupled plasma atomic emission spectroscopy (ICP) and inductively coupled plasma mass spectrometry (ICP-MS). This SOP applies to samples prepared by EPA Method 3010, with the method modifications mentioned in Table 2.

1.1 Definitions - none.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the acid digestion of aqueous samples by EPA Method 3010. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

It is the responsibility of all Katahdin technical personnel involved in the acid digestion of aqueous samples using EPA Method 3010 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their work in the appropriate lab notebook. Any deviations from the method or irregularities with the samples should also be recorded in the lab notebook and reported to the Supervisor or designated qualified data reviewer responsible for these data.

It is the responsibility of the Supervisor to ensure that technical personnel perform acid digestions in accordance with this SOP and to confirm that their work is properly documented through periodic review of the associated logbooks.

1.3 Safety

The acids used in this procedure are highly corrosive and reactive, and spiking standards contain toxic metals. The toxicity and reactivity of client samples are usually unknown, so samples should always be assumed to present a contact hazard. To reduce or eliminate exposure to potentially harmful chemicals, lab coats, gloves, and safety glasses or goggles must be worn whenever handling samples or reagents. Additional safety apparel, including face shields, rubber aprons, dust masks, and rubber shoe protectors, is available in the metals prep lab and should be worn whenever circumstances warrant.

Acids should be added to samples slowly and carefully while watching for reactions. This should be done under a hood, in case harmful fumes are evolved.

Hood sashes should be lowered as far as possible whenever beakers are being heated in the hood. Use caution when handling hot beakers.

Each qualified analyst or technician must be familiar with Katahdin Analytical Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their supervisor, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Excess spiking solutions must be emptied into the corrosive waste carboy located in the metals prep lab for subsequent appropriate disposal in accordance with the Chemical Hygiene Plan and Safety Manual.

Sample digestates should be stored for a minimum of 60 days after digestion to allow for analysis, and reanalysis if necessary. Digestates older than 60 days may be emptied into the corrosive waste carboy in the metals prep lab for subsequent appropriate disposal in accordance with the Chemical Hygiene Plan and Safety Manual.

Any other wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Chemical Hygiene Plan and Safety Manual and SOP SD-903, “Sample Disposal,” current revision.

2.0 SUMMARY OF METHOD

The aqueous sample is refluxed with nitric acid in a covered digestion vessel. Additional nitric acid is added until the color of the digestate has stabilized. After the digestate has been evaporated to a low volume, it is refluxed with hydrochloric acid and diluted to the appropriate final volume with reagent water.

Samples may be concentrated (i.e. final digestate volume less than initial sample volume) during digestion if lower detection limits are required. Volumes of reagents and spiking standards must be added in proportion to the final volume of the digestate. Because concentration of samples during digestion increases the concentrations of dissolved solids

and may exacerbate analytical interferences, concentration factors greater than 5 are not recommended.

3.0 INTERFERENCES

Interferences are discussed in the applicable analytical SOPs.

4.0 APPARATUS AND MATERIALS

4.1. 250 mL and 400 mL pre-cleaned Griffin beakers (cleaned according to the current revision of SOP CA-100, "Labware Cleaning") for digestion using a hot plate. If digestion will be performed using a block digester, 70ml graduated, polyethylene block digester tubes (with attached snap caps) will be used instead of glass beakers.

4.2 Ribbed watch glasses. If digestion is performed using a hot plate, 75 mm diameter and 100 mm diameter glass watch glasses (pre-cleaned as above) are used. If digestion is performed using a block digester, 40mm diameter disposable polyethylene watch glasses are used.

4.3 Adjustable volume automatic pipets covering the range from 10 uL to 1000 uL and disposable pipet tips; calibrated Finn pipets or Eppendorf pipets are acceptable.

4.4 Disposable graduated polystyrene specimen containers with pouring lips, 200 mL capacity.

4.5 Hot plate, block digester, or other heating source - adjustable and capable of maintaining a temperature of 90-95^{o}C. Hot plates must be numbered for easy identification.

4.6 Device for measuring hot plate temperature. This may consist of a heat-resistant 100ml beaker containing reagent water in which a thermometer is immersed. When using a block digester, a digestion tube containing reagent water in which a thermometer is immersed may be used. The temperature of one hot plate is measured each day, on a rotating basis. The hot plate identification number and the measured temperature are recorded on the sample preparation logbook sheet.

4.7 Plastic funnels, pre-cleaned as in Section 4.1.

4.8 Filter funnel holders, capable of suspending plastic funnels above disposable specimen containers.

4.9 Polyethylene wash bottles for dispensing reagent water and 5% HNO_3.

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

4.10 Filter paper, Whatman No. 41 or equivalent. Filters are acid-washed immediately prior to use as follows. Place a pre-cleaned funnel in the funnel holder and put a disposable plastic specimen container under the funnel to collect the rinsates. Place a folded filter in the funnel and rinse three times with approximate 10 mL volumes of 5% HNO_3, making sure the entire surface of the filter is wetted each time and allowing each rinse to drain completely before continuing. Then rinse three times with approximate 25 mL volumes of reagent water. Discard the rinsates into the appropriate waste container. The acid-washed filter is now ready for use.

4.11 Polyethylene sample containers with screw caps or graduated polyethylene sample containers with attached snap lids, 125 mL capacity. These are not necessary when using the block digester since the final digestates are stored in the digestion tubes.

4.12 Repipetters (adjustable repeating pipetters with reservoirs) for dispensing concentrated nitric acid and 1:1 HCl.

5.0 REAGENTS

5.1 Concentrated nitric acid, HNO_3 – trace metals grade.

5.2 Concentrated hydrochloric acid, HCl – trace metals grade.

5.3 Reagent water - water that meets the performance specifications of ASTM Type II water (ASTM D1193).

5.4 Hydrochloric acid, 1:1. Add a volume of concentrated hydrochloric acid to an equivalent volume of reagent water and swirl gently to mix.

5.5 Nitric acid, 5% v/v. Add 25 mL concentrated HNO_3 to 475 mL reagent water in a 500 mL wash bottle. Cap, point the dispensing tip into a sink, and shake gently to mix.

5.6 Multi-element spiking solutions (as listed in Figure 3).

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples to be analyzed for dissolved metals should be filtered through a 0.45 um membrane filter and preserved as soon as possible after collection. Samples to be analyzed for total metals should be preserved, unfiltered, as soon as possible after collection. Aqueous samples are preserved by acidification with nitric acid to a pH of <2.

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Please refer to Katahdin Analytical Services SOP CA-108, "Basic Laboratory Technique", current revision, for information on subsampling.

7.0 PROCEDURES

7.1 Prior to performing the digestion, make a list of the samples that are to be digested. Enter digestion information (Katahdin Sample Numbers, QC Batch ID, preparation date, analyst initials, etc.) into the ACCESS computer spreadsheet. Print out a copy of the spreadsheet. With a permamament marker, make sample labels and attach to the polyethylene sample containers that will contain the digestates.

7.2 If using glass beakers as the digestion vessels, submerge previously cleaned beakers three times into a 10% nitric acid bath, then rinse three times with reagent water. The polyethylene digestion tubes used in conjunction with the block digester do not require acid rinsing or precleaning. Label the digestion vessels with sample numbers.

7.3 If digestion is performed using a block digester, the sample aliquot may be measured in the digestion vessel using the graduations on the digestion tubes. Measure 50 ml of well-mixed sample into a 70 ml block digestion tube. A larger sample aliquot may be used (up to 250 mL) if concentration of the sample during digestion is desired. Sample volumes larger than 50 mL may be digested in 250 mL beakers. Measure aliquot of well-mixed sample into a graduated specimen cup and transfer into a properly cleaned 250 mL beaker. Sample volumes of more than 50ml may not be digested using the 70ml block digester tubes. The volumes of reagents and spiking solutions used must be adjusted in proportion to the final digestate volume. The reagent and spiking solution volumes listed below are based on a final volume of 50 mL.

7.4 Add spike solutions to matrix spike samples and laboratory control samples (refer to Figure 3 for spiking instructions).

7.5 Use a repipetter to add 1.5 mL of concentrated HNO_3 (per 50 mL final volume) to the sample. Cover with a ribbed watch glass and place on heatsource. Heat cautiously, without boiling the sample, and evaporate to a low volume (10 - 15 mL).

NOTE: Do not allow any portion of the bottom of the digestion vessel to go dry during any part of the digestion. If a sample is allowed to go to dryness, low recoveries may result. Should this occur, discard the digestate and re-prepare the sample.

7.6 Cool the sample and add another 1.5 mL aliquot (per 50 mL final volume) of concentrated HNO_3. Cover and resume heating, increasing the temperature until a gentle reflux action occurs.

7.7 Continue heating, adding additional acid as necessary, until the digestate is light in color or does not change in appearance with continued refluxing.

7.8 Evaporate digestate to a low volume (10 - 15 mL).

7.9 Cool the sample and use a repipetter to add 5 mL (per 50 mL final volume) of 1:1 HCl. Cover the sample and resume heating, refluxing for an additional 15 minutes to dissolve any precipitate or residue resulting from evaporation.

7.10 Allow the sample to cool.

7.11 If the digestate contains visible particulate material, it must be filtered. Use a pre-cleaned funnel and acid-rinsed filter paper to filter the digestate into a clean graduated plastic specimen container or block digester digestion tube. Using a wash bottle, rinse the digestion vessel with reagent water and add the rinsates to the filter apparatus. After all of the liquid in the filter has drained into the specimen container or digestion tube, thoroughly rinse the filter three times with small (5-10 mL) volumes of reagent water, allowing the liquid to drain completely after each rinse.

If the digestion was performed using hot plates and the digestate does not contain particulate material, simply decant the digestate into a clean graduated specimen container (or graduated sample container with attached snap lid), rinse the beaker with reagent water, and add the rinsates to the container.

If the digestion was performed using a block digester and the digestate contains no visible particulate material, the digestate may be brought to final volume and stored in the digestion tube without decanting or rinsing.

7.12 Using the graduations on the specimen container, snap-lid container or digestion tube, dilute to the required final volume with reagent water. If a specimen container has been used, transfer the contents to the corresponding labeled polyethylene sample bottle, cap the bottle, and discard the empty specimen container. If a snap-lid container or digestion tube has been used, close and secure the snap-lid. Shake the container gently to mix. The digestate is now ready for analysis.

7.13 Review the ACCESS computer spreadsheet for accuracy. If any information is incorrect, make the necessary changes to the computer spreadsheet and print out a corrected copy. Do not discard the original copy of the spreadsheet. Record (hand write) the sample bottle ID, reagent lot numbers, spiking information, initial and final volumes, hot plate ID and hot plate temperature in the appropriate spaces on the spreadsheet. Record any method deviations, irregularities with the samples, or other pertinent observations at the bottom of the page, and sign and date the spreadsheet. Bind all copies of the spreadsheet in the sample preparation log. An example sample preparation logbook page (ACCESS spreadsheet) is included as Figure 1.

7.14 Place each batch of digestates in a box labeled with the QC Batch ID, and put the box of digestates in the metals digestates storage area.

7.15 A condensation of the procedure described above is included in this SOP as Table 3. A controlled copy of this table may be posted in the metals preparation laboratory for reference by the analyst.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

8.1 At least one preparation blank for waters (PBW) is processed concurrently with each digestion batch of 20 or fewer samples, and is used to assess contamination resulting from the digestion procedure. The PBW consists of an aliquot of reagent water that is digested using the same reagents as those used to digest associated samples. The initial and final volumes of the PBW must be identical to those of the associated samples (i.e., if the associated samples were concentrated during digestion, the PBW must also be concentrated). Refer to the appropriate analytical SOP for PBW acceptance criteria and corrective actions.

8.2 At least one laboratory control sample for waters (LCSW) is processed concurrently with each digestion batch of 20 or fewer samples. The LCSW consists of an aliquot of reagent water that is spiked to contain all analytes of interest at known concentrations, and is digested using the same reagents as those used to digest associated samples. The initial and final volumes of the LCSW must be identical to those of the associated samples (i.e., if the associated samples were concentrated during digestion, the LCSW must also be concentrated). Directions for spiking the LCSW are contained in Figures 3 and 4. The measured analyte recoveries for the LCSW are used to assess digestion method performance. Refer to the appropriate analytical SOP for LCSW recovery acceptance criteria and corrective actions.

8.3 Matrix spiked samples are processed concurrently with each digestion batch at a minimum frequency of one per digestion batch. A matrix spike sample consists of an aliquot of a sample that is spiked with known amounts of all analytes of interest. Matrix spike recoveries are used to assess the effects of sample matrix on digestion and analysis performance. Directions for spiking matrix spike samples are contained in Figures 3 and 4. Refer to the appropriate analytical SOP for matrix spike recovery acceptance criteria and corrective actions.

8.4 Matrix spiked duplicate samples are processed concurrently with each digestion batch at a minimum frequency of one per digestion batch. Matrix spiked duplicate samples are used to assess the precision of the digestion and analysis methods. Refer to the appropriate analytical SOP for matrix spike duplicate precision acceptance criteria and corrective actions.

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

NOTE: Clients may choose specific samples for matrix spike and matrix spike duplicate analysis; otherwise, the choice is left to the person performing the digestion. The sample volumes available may restrict the choice of samples used for matrix spike and duplicate digestion. Field blank samples should not be chosen for matrix spike and matrix spike duplicate analysis.

8.5 The quality control measures and frequencies described above are minimum requirements. They are summarized for reference in Table 1. Individual clients and analytical programs may impose additional QC requirements.

9.0 METHOD PERFORMANCE

Refer to the applicable analytical SOPs for method performance information.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007, Method 3010A.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.1, 04/22/09.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

LIST OF TABLES AND FIGURES

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

TABLE 1

QC REQUIREMENTS

Analytical Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
3010	Preparation Blank for Waters (PBW)	One per prep batch of 20 or fewer samples	Refer to analytical method	Refer to analytical method
	Laboratory Control Sample for Waters (LCSW)	One per prep batch of 20 or fewer samples	Refer to analytical method	Refer to analytical method
	Matrix Spike Sample	One per prep batch	Refer to analytical method	Refer to analytical method
	Matrix Spike Duplicate Sample	One per prep batch	Refer to analytical method	Refer to analytical method
	Demonstration of analyst proficiency; accuracy and precision	One time demonstration by each analyst performing the method	Must pass all applicable QC for method	Repeat analysis until able to perform passing QC; document successful performance in personal training file

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-604-05	EPA METHOD 3010, current revision
Apparatus/Materials	1) Disposable plastic specimen cup used to measure sample volume. 2) Digestion performed in 250 mL, 400 mL Griffin beaker, or 70ml digestion tube to facilitate evaporation. 3) Ribbed watch glass used throughout digestion to reduce contamination.	1) Graduated cylinder used to measure sample volume. 2) Digestion performed in 150 mL Griffin beaker. 3) Ribbed and non-ribbed watch glasses alternated in digestion.
Procedures	1) Digestate may be analyzed for antimony and silver. 2) Sample aliquots larger or smaller than 100 mL may be used. 3) Sample evaporated to 10 - 15 mL.	1) Digestate may not be analyzed for antimony and silver. 2) Requires sample aliquot of 100 mL. 3) Sample evaporated to 5 mL.

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

TABLE 3

PROCEDURE CONDENSATION: EPA METHOD 3010

1. If performing digestion on a hot plate, rinse glass beakers and ribbed watch glasses 3 times in acid bath. Then rinse beakers and watch glasses 3 times with reagent water. If performing digestion with block digester, polyethylene digestion tubes do not require precleaning.
2. Label digestion vessels with sample numbers.
3. Mix sample well, measure 50 mL (or smaller or larger aliquot) into a polyethylene digestion tube. If using glass beakers, measure aliquot into graduated specimen container, and transfer to appropriate digestion vessel.
4. Add spike solutions to matrix spike samples and LCSW (refer to Figure 3 of this SOP).
5. Add 1.5 mL (per 50 mL final volume) concentrated HNO_3 to sample.
6. Cover with a ribbed watch glass.
7. Place on heating device (hotplate or block digester) and evaporate to 10 - 15 mL.
8. Cool sample and add another 1.5 mL (per 50 mL final volume) concentrated HNO_3.
9. Resume heating until gentle reflux action occurs.
10. Continue heating, adding additional HNO_3 as necessary until digestion is complete.
11. Evaporate to 10 - 15 mL.
12. Cool sample and add 5 mL (per 50 mL final volume) 1:1 HCl. Resume heating and reflux gently for 15 minutes.
13. Cool sample and filter (if necessary) or decant into a graduated polyetheyne digestion tube. Rinse beaker with reagent water and filter or decant rinsate into specimen container.
14. Dilute to appropriate final volume with reagent water.
15. Cap sample container and shake gently to mix.

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

EXAMPLE PAGE FROM METALS SAMPLE PREPARATION LOGBOOK

Katahdin Analytical Services, Inc. — **Metals Preparation Benchsheet**

Reagent Information:
JT Baker HNO3: H14024 JT Baker HCL: IT3907 KMG H2O2: N/A **Method:** 3010

LCS / Spike — **LCS/Spiking Information:**

☑	☑	LV. CLPP-SPK-1 (ID/Vol):	MS1404 / 0.05	mL	Hot Plate/Block ID : A
☑	☑	CLPP-SPK-INT1 (ID/Vol):	MW2021 / 0.5	mL	Start Time/Temp.: 930 / 95 °C
☑	☑	CLPP-SPK-INT2 (ID/Vol):	MW2209 / 0.5	mL	End Time/Temp. : 1500 / 95 °C
☑	☑	Uranium Spike (ID/Vol):	MS1594 / 0.005	mL	Thermometer ID/Pos.: ALC 8 / 1.4
☐	☐	CLPP-SPK-4 (ID/Vol):	N/A /	mL	

Fisher Filter Paper: 411672325 2

Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Vol	Final Units	MX	Meth	Anal.	Date	Initial Color	Initial Clarity	Final Color	Final Clarity	Artifacts	Bottle
LCSWAB011CW0	AB011CW0	0.05	L	0.05	L	AQ	IC	AJB	02/01/2010	N/A	N/A	N/A	N/A		
PBWAB011CW0	AB011CW0		L		L	AQ	IC	AJB	02/01/2010	N/A	N/A	N/A	N/A		
SD0405-001	AB011CW0		L		L	AQ	IC	AJB	02/01/2010						A
SD0405-001P	AB011CW0		L		L	AQ	IC	AJB	02/01/2010						
SD0405-001S	AB011CW0		L		L	AQ	IC	AJB	02/01/2010						
SD0405-002	AB011CW0		L		L	AQ	IC	AJB	02/01/2010						
SD0405-003	AB011CW0		L		L	AQ	IC	AJB	02/01/2010						
SD0405-004	AB011CW0		L		L	AQ	IC	AJB	02/01/2010						
SD0405-005	AB011CW0		L		L	AQ	IC	AJB	02/01/2010						
SD0422-001	AB011CW0		L		L	AQ	IC	AJB	02/01/2010						B
SD0423-001	AB011CW0		L		L	AQ	IC	AJB	02/01/2010						A
SD0429-001	AB011CW0		L		L	AQ	IC	AJB	02/01/2010						B
SD0429-002	AB011CW0		L		L	AQ	IC	AJB	02/01/2010						
SD0455-001	AB011CW0		L		L	AQ	IC	AJB	02/01/2010						
SD0455-002	AB011CW0		L		L	AQ	IC	AJB	02/01/2010						
SD0455-003	AB011CW0		L		L	AQ	IC	AJB	02/01/2010						
SD0455-004	AB011CW0		L		L	AQ	IC	AJB	02/01/2010						

AJB 2-1-10

Digestion performed by: AJB **On:** 2-1-10

Page: AB003 **Revision:** 00

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

FIGURE 2

PREPARATION OF MATRIX SPIKES, LABORATORY CONTROL SAMPLES, AND SPIKING SOLUTIONS FOR DIGESTION OF AQUEOUS SAMPLES BY METHOD 3010

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 50 mL Final Volume (mL)
Laboratory Control Sample (LCSW) and Matrix Spike	CLPP-SPK-1	Inorganic Ventures	0.050
	CLPP-SPK-INT1	Lab Prepared (see below)	0.50
	CLPP-SPK-INT2	Lab Prepared (see below)	0.50
	1000 mg/L Uranium Standard	Inorganic Ventures	0.005

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
CLPP-SPK-INT1	1000 mg/L Se	High Purity Standards	1.0
	1000 mg/L As	High Purity Standards	1.0
	1000 mg/L Pb	High Purity Standards	1.0
	1000 mg/L Cd	High Purity Standards	2.5
	1000 mg/L Sb	High Purity Standards	1.0
	10000 mg/L K	High Purity Standards	10.0
	10000 mg/L Na	High Purity Standards	7.5
	10000 mg/L Mg	High Purity Standards	5.0
	10000 mg/L Ca	High Purity Standards	2.5
CLPP-SPK-INT2	2007ICS-1	Inorganic Ventures	10.0
	1000 mg/L Sr	High Purity Standards	5.0
	1000 mg/L Sn	High Purity Standards	5.0
	10000 mg/L Si	High Purity Standards	5.0

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

FIGURE 3

ELEMENT CONCENTRATIONS IN MATRIX SPIKES, LABORATORY CONTROL SAMPLES, AND THEIR COMPONENT SPIKING SOLUTIONS FOR DIGESTION OF AQUEOUS SAMPLES BY METHOD 3010

Element	CONCENTRATION IN SOLUTION, mg/L							
	Matrix Spike	LCSW	CLPP-SPK-1	CLPP-SPK-4	CLPP-SPK-INT1	CLPP-SPK-INT2	2007 ICS-1	1000 mg/L U
Aluminum	2.000	2.000	2000					
Antimony	0.500	0.500		100	100			
Arsenic	0.500	0.500		4	10			
Barium	2.000	2.000	2000					
Beryllium	0.050	0.050	50					
Boron	0.500	0.500		50		50	500	
Cadmium	0.250	0.250		5	25			
Calcium	2.500	2.500			250			
Chromium	0.200	0.200	200					
Cobalt	0.500	0.500	500					
Copper	0.250	0.250	250					
Iron	1.000	1.000	1000					
Lead	0.500	0.500		2	10			
Magnesium	5.000	5.000			500			
Manganese	0.500	0.500	500					
Molybdenum	0.300	0.300		30		30	300	
Nickel	0.500	0.500	500					
Potassium	10.000	10.000			1000			
Selenium	0.500	0.500		5	50			
Silicon	5.230	5.230				523	230	
Silver	0.050	0.050	50					
Sodium	7.500	7.500			750			
Strontium	0.500	0.500		50		50		
Thallium	0.500	0.500		5	10			
Tin	0.500	0.500		50		50		
Titanium	1.000	1.000		100		100	1000	
Uranium	0.100	0.100						1000
Vanadium	0.500	0.500	500					
Zinc	0.500	0.500	500					

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES AND GFAA

Prepared By: George Brewer Date: 3/98

Approved By:

Group Supervisor: George Brewer Date: 01/24/01

Operations Manager: Jol C. Burton Date: 1/24/01

QA Officer: Deborah J. Nadeau Date: 1.24.01

General Manager: Duncan F. Kinsman Date: 1/25/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 3050B	Format changes, added pollution prevention, added MSD, added spiking instruction tables.	DN	1.24.01	1/24/01
02 3050B	Removed all references/procedures devoted to GFAA. Added use of digestates for ICP/MS analysis. Revised standard solution names & concs. in Tables 3 & 4 to reflect current practice.	DN	8.29.02	8.29.02
03 3050B	New Title to include ICMOS, 3. Use of digestion block and polyethylene digestion tubes added to sections 4.0, 7.0 and Table 1. PBS changed from 1.0g water to 1.0g boiling chips. H_2O_2 addition from 3.0ml then 7.0mls to 2.0ml, 2.0ml then 7.0ml. Figures and Tables updated to reflect current practices.	LAD	03/08	03/08
04	Updated Tables 3 and 4 with current spike concentrations and volumes added. Updated logbook page. Added CA-108 reference for subsampling information.	LAD	08/09	08/09

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

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STANDARD OPERATING PROCEDURE

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Recipient: _____ Date: _____

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the Katahdin Analytical Services, Inc. procedure utilized to dissolve solid matrices and solubilize metals from solid samples prior to analysis for metals by ICP-AES and ICP-MS. This SOP applies to samples prepared by EPA Method 3050, with method modifications as summarized in Table 2.

This procedure applies to all solid sample (e.g. sediments, sludges, soils, and ashes) preparations for ICP-AES and ICP-MS analyses. This method is not a total digestion technique for most samples. It is a very strong acid digestion that will dissolve almost all elements that could become "environmentally available". By design, elements bound in silicate structures are not normally dissolved by this procedure as they are not usually mobile in the environment.

1.1 Definitions

ICP-AES – Inductively Coupled Plasma Atomic Emission Spectroscopy.

ICP-MS – Inductively Coupled Plasma Mass Spectrometry.

LCSS – Laboratory Control Sample for Solids – A standard or solid reference material that has been brought through the sample preparation process.

Matrix Spike – An aliquot of a sample to which a known amount of analyte has been added before digestion.

PBS – Preparation Blank for Solids – An aliquot of reagent water that has been brought through the sample preparation process.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the acid digestion of solid samples by USEPA Method 3050 for metals analysis. Each analyst must demonstrate the ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Training".

It is the responsibility of all Katahdin technical personnel involved in the acid digestion of solid samples by USEPA Method 3050 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the procedure or irregularities with the samples should also be recorded in the lab notebook and reported to the responsible Department Manager or designated qualified data reviewer.

It is the responsibility of the Department Manager to ensure that technical personnel perform acid digestions in accordance with this SOP and to confirm that their work is properly documented through periodic review of the associated logbooks.

1.3 Safety

The acids used in this procedure are highly corrosive and reactive, and spiking standards contain toxic metals. The toxicity and reactivity of client samples are usually unknown, so samples should always be assumed to present a contact hazard. To reduce or eliminate exposure to potentially harmful chemicals, lab coats, gloves, and safety glasses or goggles must be worn whenever handling samples or reagents. Additional safety apparel, including face shields, aprons, dust masks, and shoe protectors, is available in the Metals prep lab and should be worn whenever circumstances warrant.

Acids should be added to samples slowly and carefully, while watching for reactions. This should be done under a hood, in case harmful fumes are evolved.

Hood sashes should be lowered as far as possible whenever beakers are being heated on a hot plate. Use caution when handling hot beakers.

Personnel are required to read the Katahdin Hazrdous Waste Management Plan and Safety Manual before performing this procedure, and must be familiar with the general rules for laboratory safety, personal hygiene, housekeeping, and use of protective clothing and equipment.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Excess spiking solutions must be emptied into the corrosive waste carboy located in the Metals prep lab for subsequent appropriate disposal in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual.

Sample digestates should be stored for a minimum of 60 days after digestion to allow for analysis, and reanalysis if necessary. Digestates older than 60 days may be emptied into the corrosive waste carboy in the Metals prep lab for subsequent appropriate disposal in accordance with the Katahdin Hazardous Waste Mnagement Plan and Safety Manual.

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

2.0 SUMMARY OF METHOD

A representative 1 to 2 g (wet weight) sample is digested with repeated additions of nitric acid and hydrogen peroxide. Hydrochloric acid is added to the initial digestate and the sample is refluxed. The digestate is then filtered and diluted to a final volume of 100 mL.

3.0 INTERFERENCES

Interferences are discussed in the applicable analytical SOPs.

4.0 APPARATUS AND MATERIALS

4.1 Digestion vessels. If digestion is performed using a hot plate, the appropriate digestion vessels are 100 mL pre-cleaned Griffin beakers (cleaned according to the current revision of SOP CA-100, "Labware Cleaning" and CA-602, “Glassware Preparation and Sample Preservation for Trace Element Analyses”). If digestion is performed using a block digester, the appropriate digestion vessels are new 70 mL disposable graduated polyethylene digestion tubes with attached snap lids.

4.2 Ribbed watch glasses. If digestion is performed using a hot plate, 75 mm diameter glass watch glasses (pre-cleaned as above) are used. If digestion is performed using a block digester, 40 mm diameter disposable polyethylene watch glasses are used.

4.3 Adjustable volume automatic pipets covering the range from 10 uL to 1000 uL and disposable pipet tips; calibrated Finn pipets or Eppendorf pipets are acceptable.

4.4 Disposable graduated polystyrene specimen containers with pouring lips, 200 mL capacity.

4.5 Hot plate or block digester, griddle, or other heating source - adjustable and capable of maintaining a temperature of $95^{o}C \pm 5^{o}C$. Heating sources must be numbered for easy identification.

4.6 Device for measuring hot plate temperature, consisting of a flask or digestion vessel in which the bulb of a thermometer is immersed in sand or water. The temperature of each hot plate used is measured and recordedeach day. The hot plate identification number and the measured temperature are recorded on the sample preparation logbook sheet.

4.7 Plastic funnels, pre-cleaned as in Section 4.1.

4.8 Filter funnel holders, capable of suspending plastic funnels above disposable specimen containers.

4.9 Polyethylene wash bottles for dispensing reagent water and 5% HNO_3.

4.10 Filter paper, Whatman No. 41 or equivalent. Filters are acid-washed immediately prior to use as follows. Place a pre-cleaned funnel in the funnel holder and put a disposable plastic specimen container under the funnel to collect the rinsates. Place a folded filter in the funnel and rinse three times with approximate 10 mL volumes of 5% HNO_3, making sure the entire surface of the filter is wetted each time and allowing each rinse to drain completely before continuing. Then rinse three times with approximate 25 mL volumes of reagent water, again allowing each rinse to drain completely. Discard the rinsates into the appropriate waste container. The acid-washed filter is now ready for use.

4.11 Polyethylene sample containers with screw caps or graduated polyethylene sample containers with attached snap lids, 125 mL capacity.

4.12 Repipetters (adjustable repeating pipetters with reservoirs) for dispensing concentrated nitric acid, 1:1 HNO_3, and concentrated HCl.

4.13 Analytical balance capable of reading to 0.01 gram.

4.14 Spatulas, scoops, or spoons; plastic or stainless steel, rinsed with 5% HNO_3 and reagent water. Disposable tongue depressors may be used and do not requrire to be rinsed.

5.0 REAGENTS

5.1 Concentrated nitric acid, HNO_3 – trace metals grade.

5.2 Concentrated hydrochloric acid, HCl – trace metals grade.

5.3 Reagent water - water that meets the performance specifications of ASTM Type II water (ASTM D1193).

5.4 Nitric acid, 1:1. Add a volume of concentrated HNO_3 to an equivalent volume of reagent water and swirl gently to mix.

5.5 Nitric acid, 5% v/v. Add 25 mL concentrated HNO_3 to 475 mL reagent water in a 500 mL wash bottle. Cap, point the dispensing tip into a sink, and shake gently to mix.

5.6 30% hydrogen peroxide (H_2O_2) - spectrometric grade.

5.7 Multielement spiking solutions (see Table 3 for a list of required spiking solutions).

5.8 Solid reference material – a soil containing all the elements of interest, with empirically established method-specific recoveries and acceptance limits for all analytes. Solid reference materials are purchased with documentation of analysis provided by the vendor. See Figure 4 for an example certificate of analysis for a solid reference material.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples should be collected in clean plastic or glass containers. Samples must be refrigerated ($4^oC \pm 2^oC$) upon receipt by the laboratory. The holding time for solid samples is 6 months from the date of sample collection.

7.0 PROCEDURE

The procedure described below is condensed for quick reference in Table 3.

SAMPLE PREPARATION

7.1 Prior to performing the digestion, make a list of the samples that are to be digested. Enter digestion information (Katahdin Sample Numbers, QC Batch ID, preparation date, analyst initials, etc.) into the ACCESS computer spreadsheet. Print out a copy of the spreadsheet (see Figure 2 for an example). Hand label the digestate vessels

7.2 If using glass beakers as the digestion vessels, submerge previously cleaned beakers and watch glasses three times into a 10% nitric acid bath, then rinse three times with reagent water. The polyethylene digestion tubes used in conjunction with the block digeter do not require acid rinsing or precleaning. Label the digestion vessels with sample numbers.

7.3 Weigh 1 to 2 g of well-mixed sample into a properly cleaned, labeled, and tared Griffin beaker or polyethylene digestion tube. Record (hand write) the weight of each sample on the printout of the digestion spreadsheet. Refer to Katahdin Analytical Services SOP CA-108, current revision “Basic Laboratory Technique” for more information on subsampling.

7.4 Weigh an appropriate amount of solid reference material to a clean, labeled, and tared Griffin beaker or polyethylene digestion tube to serve as a laboratory control sample.

7.5 Add spike solutions to matrix spike samples (refer to Tables 3 and 4 for spiking instructions).

7.6 Using repipetters, add 10 mL of 1:1 HNO_3, mix the slurry. Cover with a ribbed watch glass and place on heat source. Gently heat the sample to 95^{o}C ± 5 ^{o}C and reflux for 10 to 15 minutes without boiling. Remove the digestion vessel from the heat source and cool the sample.

7.7 Add 5 mL of concentrated HNO_3 to the sample, replace the watch glass, and reflux for 30 minutes. If brown fumes are generated, indicating oxidation of the sample by HNO_3, repeat this step (addition of 5 mL of concentrated HNO_3) until no brown fumes are given off by the sample, indicating complete reaction by HNO_3.

7.8 Continue heating the sample at 95^{o}C ± 5^{o}C without boiling until the digestate has evaporated to approximately 5 to 10 mL or until two hours have elapsed, whichever occurs first. Do not allow the sample to go to dryness. Remove the digestion vessel from the heat source and cool the sample.

7.9 Add 2 mL of reagent water and 2 mL of 30% H_2O_2 to the sample, replace the watch glass, and heat gently on the heat source to start the peroxide reaction. Continue heating until effervescence subsides.

7.10 Add an additional 2 mL of 30% H_2O_2 to the sample, replace the watch glass, and heat gently on the heat source to start the peroxide reaction. Continue heating until effervescence subsides.

7.11 Add an additional 6 mL of 30% H_2O_2 in 1-mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged.

7.12 Continue heating the sample at 95^{o}C ± 5^{o}C without boiling until the digestate has evaporated to approximately 5 to 10 mL or until two hours have elapsed, whichever occurs first. Do not allow the sample to go to dryness. Remove the sample from the heat source and cool.

7.13 Add 10 mL of concentrated HCl to the digest from 7.12, replace the watch glass, and reflux at 95^{o}C ± 5^{o}C for 15 minutes. Remove the sample from the heat source and cool.

7.14 Use a pre-cleaned funnel and acid-rinsed filter paper to filter the digestate into a clean graduated polystyrene specimen container or graduated polyethylene sample container with attached snap lid. Using a wash bottle, rinse the digestion vessel with reagent water and add the rinsates to the filter apparatus. After all of the liquid in the filter has drained into the specimen container, thoroughly rinse the filter three times with small (5-10 mL) volumes of reagent water, allowing the liquid to drain completely after each rinse. Using the graduations on the specimen container or snap-lid container, dilute to 100 mL with reagent water. If a specimen container has been used, transfer the contents to the corresponding labeled polyethylene sample bottle, cap the bottle, and discard the empty specimen container. If a snap-lid

container has been used, close and secure the snap-lid. Shake the container gently to mix. The digestate is now ready for ICP-AES or ICP-MS analysis.

7.15 Review the ACCESS computer spreadsheet for accuracy. If any information is incorrect, make the necessary changes to the computer spreadsheet and print out a corrected copy. Do not discard the original copy of the spreadsheet. Record (hand write) reagent lot numbers, spiking information, and heat source temperature in the appropriate spaces on the spreadsheet. Record any method deviations, irregularities with the samples, or other pertinent observations at the bottom of the page, and sign and date the spreadsheet. Bind all copies of the spreadsheet in the sample preparation log. An example sample preparation logbook page (ACCESS spreadsheet) is included as Figure 2.

7.15 Reopen the electronic ACCESS spreadsheet for the digestion and transcribe the sample weights from the handwritten, bound copy into the electronic copy. The information in this electronic spreadsheet will later be imported into the ACCESS metals database and used to calculate sample concentrations on a weight basis.

7.16 Place each batch of digestates in a box labeled with the QC Batch ID, and put the box of digestates in the metals digestates storage area.

CALCULATIONS

7.17 Analytical results for solid samples are reported on a dry weight basis. Total solids are determined by the Wet Chemistry Group, and are recorded in spreadsheets that are electronically imported into the Access metals database. Final dry weight concentrations are calculated by the Access database as follows:

$$\text{Concentration (mg/kg dry weight)} = (C \times V) / (W \times S)$$

where: C = Measured concentration (mg/L)
V = Digestate final volume (L)
W = Sample wet weight (kg)
S = % Solids/100

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 3050 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and

standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgements. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The Depatment Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

8.1 At least one preparation blank for soils (PBS) is processed concurrently with each digestion batch of 20 or fewer samples, and is used to assess contamination resulting from the digestion procedure. The PBS consists of a 1.0 g of boiling stones that is digested using the same reagents as those used to digest associated samples. Refer to the appropriate analytical SOP for PBS acceptance criteria and corrective actions.

8.2 At least one laboratory control sample for soils (LCSS) is processed concurrently with each digestion batch of 20 or fewer samples. The LCSS consists of an aliquot of a solid reference material for which the concentrations of the analytes of interest have been empirically established (solid-matrix LCSS), or an aliquot of reagent water that is spiked to contain all analytes of interest at known concentrations (aqueous-matrix LCSS). The solid reference material should normally be used as the LCSS, unless a particular client or analytical program requires that spiked reagent water be used. The LCSS is digested using the same reagents as those used to digest associated samples. Directions for spiking the aqueous-matrix LCSS are contained in Table 3. The measured analyte recoveries for the LCSS are used to assess digestion method performance. Refer to the appropriate analytical SOP for LCSS recovery acceptance criteria and corrective actions.

8.3 Matrix spike samples are processed along with each digestion batch at a minimum frequency of one per digestion batch. A matrix spike sample consists of an aliquot of a sample that is fortified with known amounts of all analytes of interest prior to digestion. Matrix spike recoveries are used to assess the biasing effects of sample matrix on digestion and analysis performance. Directions for spiking matrix spike samples are contained in Figure 2. Refer to the appropriate analytical SOP for matrix spike recovery acceptance criteria and corrective actions.

8.4 Matrix spiked duplicate samples are processed concurrently with each digestion batch at a minimum frequency of one per digestion batch. Matrix spiked duplicate samples are used to assess the precision of the digestion and analysis methods. Refer to the appropriate analytical SOP for matrix spike duplicate precision acceptance criteria and corrective actions.

NOTE: Clients may choose specific samples for matrix spike and duplicate analysis; otherwise, the choice is left to the person performing the digestion.

8.5 The quality control measures and frequencies described above are minimum requirements. Individual clients and analytical programs may impose additional QC requirements.

9.0 METHOD PERFORMANCE

Refer to the applicable instrumental analysis SOP for method performance information.

10.0 APPLICABLE DOCUMENTS/REFERENCES

"Test Methods for the Evaluation of Solid Waste," United States Environmental Protection Agency, SW-846, Third Edition, Final Update III, 12/96, Method 3050B.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.1, 04/22/09.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

LIST OF TABLES AND FIGURES

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

TABLE 1

QC REQUIREMENTS – METHOD 3050

Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
3050	Preparation Blank for Solids (PBS)	One per prep batch of 20 or fewer samples.	Refer to analytical method.	Refer to analytical method.
	Laboratory Control Sample for Solids (LCSS)	One per prep batch of 20 or fewer samples.	Refer to analytical method.	Refer to analytical method.
	Matrix Spike Sample	One per prep batch.	Refer to analytical method.	Refer to analytical method.
	Matrix Spike Duplicate Sample	One per prep batch.	Refer to analytical method.	Refer to analytical method.
	Demonstration of analyst proficiency	One-time demonstration by each analyst performing the method.	Must pass all applicable QC for method.	Repeat analysis until able to perform passing QC; document successful performance in personal training file.

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

TABLE 2

SUMMARY OF METHOD MODIFICATIONS – METHOD 3050

Topic	Katahdin SOP CA-605-02	Method 3050, current revision
Apparatus /Materials	1) Digestion performed in 100 mL Griffin beaker or 70 mL polyethylene tube. 2) Graduated disposable plastic cup or 120 mL polyethylene tube used to bring digestate to final volume.	1) Digestion performed in 250 mL Griffin beaker. 2) Volumetric flask used to bring digestate to final volume.
Procedure	1) Digestate volume reduced to 5 to 10 mL prior to filtering. 2) After filtration, the filters are rinsed three times with reagent water. 3) 30% H_2O_2 is added in two 2 mL aliquots and then six 1 mL aliquots.	1) Digestate volume reduced to 5 mL prior to filtering. 2) After filtration, the filters are rinsed twice with reagent water. 3) 30% H_2O_2 is added in one 3 mL aliquot and then seven 1 mL aliquots.

TABLE 3

PREPARATION OF MATRIX SPIKES AND SPIKING SOLUTIONS FOR DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
Matrix Spike for ICP-AES	CLPP-SPK-1	Inorganic Ventures	0.10
	CLPP-SPK-INT1	Lab Prepared (see below)	1.00
	CLPP-SPK-INT2	Lab Prepared (see below)	1.00
	1000 mg/L Uranium Std.	High Purity Standards	0.01

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
CLPP-SPK-INT1	1000 mg/L Se	High Purity Standards	5.0
	1000 mg/L As	High Purity Standards	5.0
	1000 mg/L Pb	High Purity Standards	5.0
	1000 mg/L Cd	High Purity Standards	2.5
	1000 mg/L Sb	High Purity Standards	5.0
	1000 mg/L K	High Purity Standards	10.0
	1000 mg/L Na	High Purity Standards	7.5
	1000 mg/L Mg	High Purity Standards	5.0
	1000 mg/L Ca	High Purity Standards	2.5
CLPP-SPK-INT2	2007ICS-1	Inorganic Ventures	10.0
	1000 mg/L Sr	High Purity Standards	5.0
	1000 mg/L Sn	High Purity Standards	5.0
	10000 mg/L Si	High Purity Standards	5.0

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

TABLE 4

ELEMENT CONCENTRATIONS IN ICP-AES MATRIX SPIKES AND THEIR COMPONENT SPIKING SOLUTIONS FOR DIGESTION OF SOLID SAMPLES BY METHOD 3050

Element	CONCENTRATION IN SOLUTION, mg/L							
	Matrix Spike (ICP-AES)	CLPP-SPK-1	CLPP-SPK-4	CLPP-SPK-INT1	CLPP-SPK-INT2	QCP-CICV-3 SPK-3	2007 ICS- 1	1000 mg/L U
Aluminum	2.000	2000						
Antimony	0.500		100	50				
Arsenic	0.500		4	50		500		
Barium	2.000	2000						
Beryllium	0.050	50						
Boron	0.500		50		50		500	
Cadmium	0.250		5	25		250		
Calcium	2.500			250				
Chromium	0.200	200						
Cobalt	0.500	500						
Copper	0.250	250						
Iron	1.000	1000						
Lead	0.500		2	50		500		
Magnesium	5.000			500				
Manganese	0.500	500						
Molybdenum	0.300		30		30		300	
Nickel	0.500	500						
Potassium	10.000			1000				
Selenium	0.500		5	50		500		
Silicon	5.230				523		230	
Silver	0.050	50						
Sodium	7.500			750				
Strontium	0.500		50		50			
Thallium	0.500		5	250		500		
Tin	0.500		50		50			
Titanium	1.000		100		100		1000	
Uranium	0.100							1000
Vanadium	0.500	500						
Zinc	0.500	500						

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

FIGURE 1

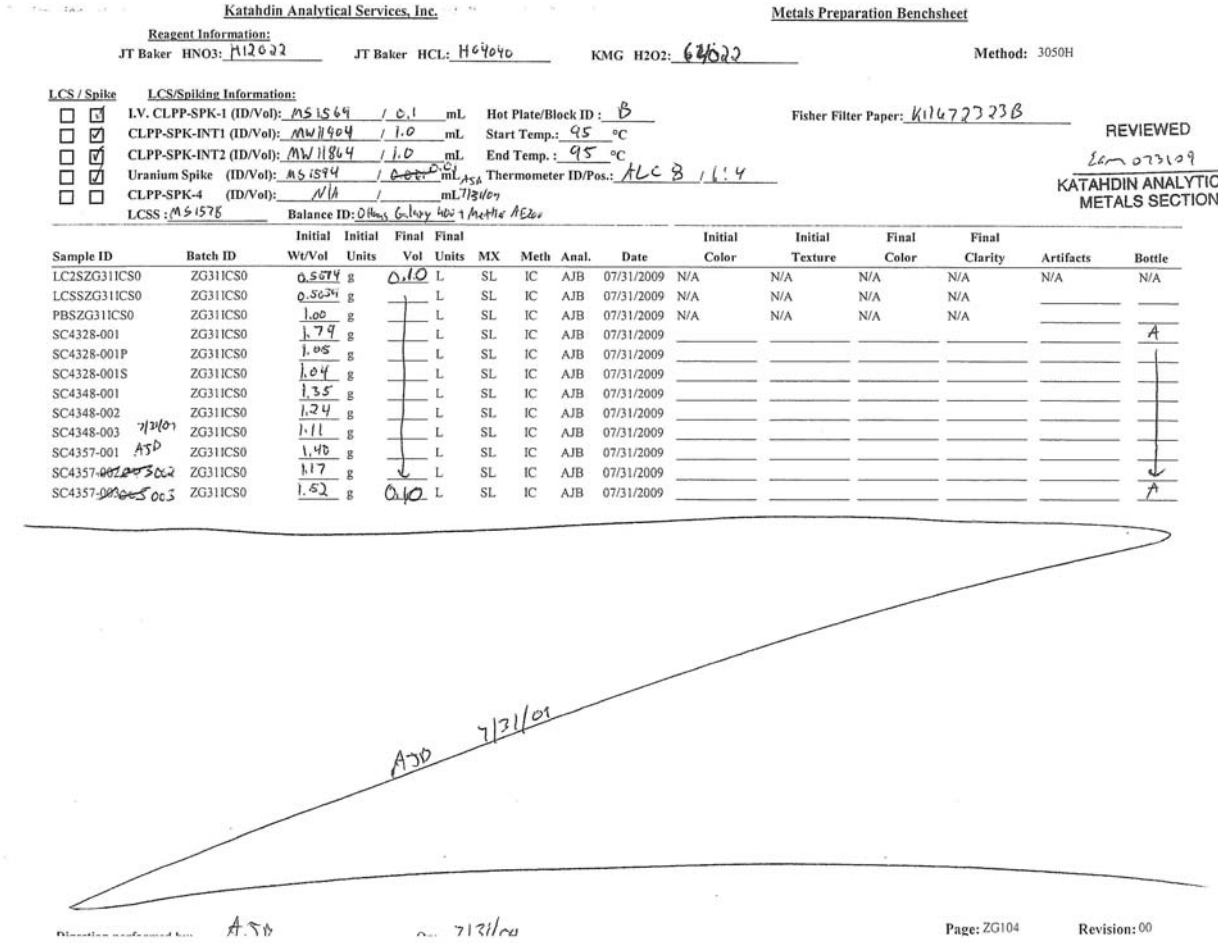
PROCEDURE CONDENSATION – METHOD 3050

1. Prepare and print out ACCESS spreadsheet.
2. If performing digestion on a hot plate, rinse 250 mL Griffin beakers and ribbed watch glasses 3 times in acid bath. Then rinse beakers and watch glasses 3 times with laboratory reagent grade water. If perfoming digestion with block digester, polyethylene digestion tubes do not require precleaning.
3. Label digestion vessels (beakers or polyethylene sample tubes) with sample numbers.
4. Weigh 1 to 2 g of well-mixed sample into tared digestion vessels. Record sample weights.
5. Add spike solutions to matrix spike samples.
6. Add 10 mL 1:1 HNO_3 to samples and cover with watch glasses.
7. Reflux for 10 to 15 minutes at $95^{o} \pm 5^{o}$ C. without boiling. Cool samples.
8. Add 5 mL conc. HNO_3, cover beakers, and reflux for 30 minutes.
9. Repeat Step 8 as necessary until digestion is complete.
10. Reduce sample volumes to 5 to 10 mL or heat for 2 hours, whichever occurs first.
11. Cool sample and add 2 mL reagent water and 2 mL 30% H_2O_2. Heat gently until effervescence subsides.
12. Cool sample and add 2 mL 30% H_2O_2. Heat gently until effervescence subsides.
13. Cool samples and add 6 mL of 30% H_2O_2 in 1 mL aliquots. Heat gently until effervescence subsides.
14. Reduce sample volumes to 5 to 10 mL or heat for 2 hours, whichever occurs first.
15. Add 10 mL conc. HCl and reflux for 10 to 15 minutes at $95^{o} \pm 5^{o}$ C.
16. Cool sample and filter into graduated specimen container. Bring to volume with reagent water and transfer to labeled polyethylene bottle.
17. Enter sample weights into ACCESS spreadsheet.

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

FIGURE 2

EXAMPLE PAGE FROM METALS SAMPLE PREPARATION LOGBOOK



Katahdin Analytical Services, Inc. — Metals Preparation Benchsheet

Reagent Information: JT Baker HNO3: M12022 JT Baker HCL: H64040 KMG H2O2: 6/1/22 Method: 3050H

LCS / Spike — LCS/Spiking Information:

- LV. CLPP-SPK-1 (ID/Vol): MS1569 / 0.1 mL — Hot Plate/Block ID: B — Fisher Filter Paper: K1167223B
- CLPP-SPK-INT1 (ID/Vol): MW11904 / 1.0 mL — Start Temp.: 95 °C
- CLPP-SPK-INT2 (ID/Vol): MW11864 / 1.0 mL — End Temp.: 95 °C
- Uranium Spike (ID/Vol): MS1594 / ~~0.02~~ 0.01 mL ASA — Thermometer ID/Pos.: ALC 8 / 1.4
- CLPP-SPK-4 (ID/Vol): N/A / mL 7/31/09
- LCSS: MS1578 — Balance ID: Ohaus Galaxy 400 / Mettler AE200

REVIEWED Em 073109 KATAHDIN ANALYTICAL METALS SECTION

Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Vol	Final Units	MX	Meth	Anal.	Date	Initial Color	Initial Texture	Final Color	Final Clarity	Artifacts	Bottle
LC2SZG311CS0	ZG311CS0	0.5074	g	0.10	L	SL	IC	AJB	07/31/2009	N/A	N/A	N/A	N/A	N/A	N/A
LCSSZG311CS0	ZG311CS0	0.5034	g		L	SL	IC	AJB	07/31/2009	N/A	N/A	N/A	N/A		
PBSZG311CS0	ZG311CS0	1.00	g		L	SL	IC	AJB	07/31/2009	N/A	N/A	N/A	N/A		
SC4328-001	ZG311CS0	1.79	g		L	SL	IC	AJB	07/31/2009						A
SC4328-001P	ZG311CS0	1.05	g		L	SL	IC	AJB	07/31/2009						
SC4328-001S	ZG311CS0	1.04	g		L	SL	IC	AJB	07/31/2009						
SC4348-001	ZG311CS0	1.35	g		L	SL	IC	AJB	07/31/2009						
SC4348-002	ZG311CS0	1.24	g		L	SL	IC	AJB	07/31/2009						
SC4348-003 7/31/09	ZG311CS0	1.11	g		L	SL	IC	AJB	07/31/2009						
SC4357-001 AJD	ZG311CS0	1.40	g		L	SL	IC	AJB	07/31/2009						
SC4357-~~002~~003	ZG311CS0	1.17	g		L	SL	IC	AJB	07/31/2009						
SC4357-~~003~~005	ZG311CS0	1.52	g	0.10	L	SL	IC	AJB	07/31/2009						A

AJD 7/31/09

Digestion performed by: AJD Date: 7/31/09 Page: ZG104 Revision: 00

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

FIGURE 3

EXAMPLE CERTIFICATE OF ANALYSIS FOR SOLID REFERENCE MATERIAL

ENVIRONMENTAL RESOURCE ASSOCIATES®
The Industry Standard™

M51475

DataPacK™
Lot No. D051-540

Trace Metals in Soil
Catalog No. 540

Certification

Method 3050 HNO3, H2O2, HCl Parameter	Total Concentration [1] (mg/Kg)	Certified Value [2] (mg/Kg)	Performance Acceptance Limits™ [3] (mg/Kg)
aluminum	55600*	7870	4630 - 11100
antimony	160	70.5	D.L. - 149
arsenic	316	289	234 - 344
barium	869	211	174 - 247
beryllium	60.9	54.4	45.2 - 63.6
boron	129	91.3	58.8 - 124
cadmium	114	101	82.9 - 119
calcium	9750*	3680	2970 - 4390
chromium	249	224	180 - 268
cobalt	113	101	82.7 - 119
copper	94.9	88.0	73.3 - 103
iron	24400*	15700	6610 - 24900
lead	184	158	129 - 187
magnesium	3780*	2260	1760 - 2750
manganese	703	420	343 - 497
mercury	5.32	5.18	3.42 - 6.87
molybdenum	80.2	69.6	55.5 - 83.7
nickel	137	120	99.1 - 141
potassium	33000*	3000	2200 - 3800
selenium	146	130	101 - 159
silver	127	104	68.9 - 139
sodium	15600*	1080	692 - 1470
strontium	326	113	90.5 - 135
thallium	106	94.0	72.8 - 115
tin	175	149	104 - 194
titanium	3100*	284	116 - 453
vanadium	151	111	85.1 - 137
zinc	311	272	215 - 329

Method 3050 HNO3, H2O2 Parameter	Total Concentration [1] mg/Kg	Certified Value [2] mg/Kg	Performance Acceptance Limits™ [3] mg/Kg
aluminum	55600*	7380	4440 - 10300
antimony	160	75.2	D.L. - 198
arsenic	316	284	225 - 343
barium	869	217	177 - 257
beryllium	60.9	53.6	42.7 - 64.5
boron	129	89.5	58.9 - 120
cadmium	114	103	83.6 - 122
calcium	9750*	3540	2800 - 4270
chromium	249	224	172 - 275
cobalt	113	101	82.0 - 120
copper	94.9	85.5	70.4 - 100
iron	24400*	12500	5480 - 19500
lead	184	162	132 - 192
magnesium	3780*	2160	1650 - 2670
manganese	703	415	330 - 500
mercury	5.32	5.18	3.42 - 6.87
molybdenum	80.2	68.8	52.7 - 84.9
nickel	137	119	98.5 - 140
potassium	33000*	2840	2160 - 3520
selenium	146	135	104 - 166
silver	127	107	49.8 - 164
sodium	15600*	1010	709 - 1310
strontium	326	111	89.0 - 133
thallium	106	99.3	76.8 - 122
tin	175	148	70.6 - 225
titanium	3100*	283	104 - 463
vanadium	151	104	70.5 - 138
zinc	311	275	222 - 328

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-608
Revision History
Cover Page
Page 1

TITLE: TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010

Prepared By: George Brewer Date: 7/98

Approved By:

Group Supervisor: George Brewer Date: 01/23/01

Operations Manager: Jol C. Burton Date: 1/23/01

QA Officer: Deborah J. Nadeau Date: 1·23·01

General Manager: Duncan F. Hufstetler Date: 1/25/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 6010B	Format changes, added pollution prevention, expanded procedure and QC sections. Added tables.	DH	1·23·01	1/23/01
02 6010B	Calibration begins with analysis of S0 (cal. blank) followed by S1 (Mixed Cal. Std.) Changes to section 7.5 and Table 8 to reflect this. Made changes to element concs. in Tables 3, 4, 5, 6 to reflect current practices.	DH	10·21·02	10·21·02
03 6010B	Added MN_IEC to standards run. Changed frequency of LRS. Changed concentration % HNO_3 in calibration blank. CRI changed from three separate solutions to one. Changed CRI vendor.	MRC	04.15.04	04.15.04
04	Updated ICV, CCV, ICB, PQL Chk std. PBW, PBS, MS & MSD acceptance criteria updated Table 1	LAD	05/06	05/06
05	Updated Tables 3, 4, 5, 6 and 7 with current standard concentrations and prep. Updated Table 1 with current practices including NAVY audit findings. Updated sections 2, 7.2, 7.6 and Table 1 with new ICP information. Updated Table 8 with current sequence requirements.	LAD	07/07	07/07

TITLE: TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Added hardness definition and calculation (APP. I)	LAD	09/07	09/07
07	Updated Summary to reflect new ICP functions. Removed ICP set-up Updated tables to reflect changes in Standard Concentrations and preparation	LAD	11/08	11/08
08	Updates to Sections 8 and 10, Tables 1 and 2 to reflect changes from 6010B to 6010C. Added LLQC information and criteria to Sect. 8 and Table 1. Added criteria to analyze PQL standard at the beginning and END of each run.	LAD	02/09	02/09
09	Updated Sections 8, 9, 10 and Table 1 for compliance with DoD QSM version 4.1.	LAD	08/09	08/09
10	Added Table 2 - DoD QSM Ver. 4.1 QC Requirements. Minor correction to Table 1.	LAD	04/10	04/10

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

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TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

1.0 SCOPE AND APPLICATION

Inductively coupled plasma atomic-emission spectroscopy (ICP-AES) determines trace elements, including metals, in solution. The purpose of this SOP is to describe the procedures used by Katahdin Analytical Services, Inc. personnel to analyze aqueous and solid samples for trace metals by USEPA Method 6010 (Test Methods for Evaluating Solid Waste, Physical/ Chemical Methods, USEPA SW846).

Sample types that may be analyzed using these methods include drinking waters, ground waters, aqueous samples, TCLP, SPLP and EP Toxicity extracts, industrial and organic wastes, soils, sludges, sediments, and other solid wastes. The following elements may be analyzed under this SOP: Al, Sb, As, Ba, Be, B, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Mo, Ni, K, Se, Si, Ag, Na, Sn, Sr, Tl, Ti, V, and Zn.

All samples, except filtered ground water samples, analyzed under USEPA Method 6010 require digestion prior to analysis. USEPA Methods 3005, 3010, and 3050 describe appropriate digestion procedures for samples to be analyzed by ICP-AES under EPA Method 6010. Refer to current revisions of Katahdin SOPs CA-604 and CA-605, current revisions, for sample digestion procedures.

1.1 Definitions

Analytical Spike - An aliquot of a sample to which a known amount of analyte has been added before analysis and after digestion, if digestion is required.

CCB - Continuing Calibration Blank - An analyte-free solution consisting of acidified reagent water used to verify calibration accuracy periodically during analysis.

CCV - Continuing Calibration Verification - A midrange standard used to verify calibration accuracy periodically during analysis.

CRI - Contract Required detection limit sample for ICP - A low concentration standard used to verify calibration accuracy near the low end of the calibration range.

Duplicate - A second aliquot of a sample that is prepared and analyzed in the same way as the original sample in order to determine the precision of the method.

ICB - Initial Calibration Blank - An analyte-free solution consisting of acidified reagent water used to verify calibration accuracy.

ICP-AES - Inductively Coupled Plasma Atomic Emission Spectroscopy.

ICS - Interference Check Sample - Two standards (ICSA and ICSAB) used to verify the effectiveness of interelement correction and background correction. Solution ICSA contains only interferents (Al, Ca, Fe, and Mg) at high concentrations (200 to

500 mg/L); solution ICSAB contains interferents at the same concentrations as well as analytes at low (20 mg/L or less) concentrations.

ICV - Initial Calibration Verification - A standard made from a source independent from the calibration standards and with analyte concentrations different from those in the CCV; used to verify the accuracy of the instrument calibration.

IDL - Instrument Detection Limit - The lowest concentration of an analyte that can be determined with 99% confidence.

LOD – Limit of Detection – An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix-specific and is used for DoD QSM acceptance criteria.

LOQ – Limit of Quantitation.- The minimum concentration of a target analyte that produces a quantitative result within specified limits of precision and bias.

LCS - Laboratory Control Sample - A standard or solid reference material that has been brought through the sample preparation process.

LRS - Linear Range Standard - A high-concentration standard used to determine the upper reporting limit of the ICP calibration.

PB - Preparation Blank - Reagent water that has been brought through the sample preparation process.

PQL - Practical Quantitation Limit - The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the IDL.

Matrix Spike - An aliquot of a sample to which a known amount of analyte has been added before digestion.

Serial Dilution - The dilution of a sample by a factor of five. When corrected by the dilution factor, the measured analyte concentrations of the diluted sample should agree with those of the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferents.

Hardness – The sum of the calcium and magnesium concentrations, both expressed as calcium carbonate, in mg/L.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of, analysts experienced in ICP analysis by EPA Method 6010. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

It is the responsibility of all Katahdin technical personnel involved in ICP analysis by Method 6010 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

Samples, sample digestates, standards, and other reagents used in ICP analysis may contain high concentrations of acids and toxic metals. Safety glasses should be worn when changing or adjusting argon tanks.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Wastes from ICP analysis should be disposed of in a manner appropriate to the hazards they present. Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Analytical Environmental Health and Safety Manual I and SOP SD-903, “Sample Disposal,” current revision. Expired

standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

2.0 SUMMARY OF METHOD

This method describes multielemental determinations by ICP-AES using simultaneous optical systems and radial and axial viewing of the plasma. The basis of the method is the measurement of atomic emission from sample atoms entrained in an argon plasma by optical spectroscopy. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where thermal excitation of entrained atoms and ions occurs. Characteristic atomic-line and ionic-line emission spectra are produced by a radio-frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating and the intensities of the emitted lines are monitored by a solid state charge injection device (CID) camera system. Photocurrents from the CID camera system are measured by a computer system. Element concentrations of unknown samples are quantitated by comparison of sample emission intensities to emission intensities of standards of known concentration. A background correction technique is used to compensate for variable background contribution to the determination of trace elements. Background is measured adjacent to the analyte lines on samples during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, has been determined by the complexity of the spectrum adjacent to the analytical line. The position used must be relatively free of spectral interference and must reflect the same change in background intensity as occurs at the analyte wavelength. Physical interferences are corrected through the use of an internal standard (yttrium) that is automatically added to all samples and standards prior to nebulization. The possibility of additional interferences (noted in section 3) must be recognized and appropriate corrections applied.

3.0 INTERFERENCES

Several types of interference effects may contribute to inaccuracies in the determination of trace elements. They can be summarized as spectral interferences, physical interferences, and chemical interferences.

Spectral interferences can be categorized as 1) overlap of a spectral line from another element; 2) unresolved overlap of molecular band spectra; 3) background contribution from continuous or recombination phenomena; and 4) background from stray light from the line emission of high concentration elements. The first of these effects is compensated by utilizing the computer correction of raw data, requiring the monitoring and measurement of the interfering element (interelement correction). The second effect is controlled by choosing analytical wavelengths that are free from overlapping molecular emission spectra. The third and fourth effects are usually compensated by a background correction adjacent to the analyte line. Uncorrected spectral interferences may be detected through examination of serial dilution and matrix spike data.

Physical interferences are generally considered to be effects associated with sample nebulization and transport processes. Such properties as changes in viscosity and surface tension can cause significant inaccuracies, especially in samples that may contain high dissolved solids and/or acid concentrations. Matrix matching of standards and samples and the use of a peristaltic pump may lessen these interferences. If these types of interferences are operative, they must be reduced by dilution of the sample and/or utilization of standard addition techniques. Another problem that can occur from high dissolved solids is salt buildup at the tip of the nebulizer. This affects aerosol flow rate causing instrumental drift. Regular cleaning of nebulizer tips and dilution of samples with high dissolved solids contents are used to control this problem. Physical interferences are also corrected by this laboratory through the use of an internal standard. Uncorrected physical interferences may be detected through examination of serial dilution and matrix spike data. Instrument drift caused by the salting up of nebulizer tips may also be detected by looking for oriented drift in calibration verification standards analyzed regularly throughout the run.

Chemical interferences are characterized by molecular compound formation, ionization effects, and solute vaporization effects. Normally these effects are not pronounced with the ICP technique; however, if observed they can be minimized by careful selection of operating conditions (i.e., incident power, observation position, etc.), by matrix matching, and by standard addition procedures. These types of interferences can be highly dependent on matrix type and the specific analyte element. Uncorrected chemical interferences may be detected through examination of serial dilution data.

4.0 APPARATUS AND MATERIALS

4.1 Computer-controlled inductively-coupled plasma atomic emission spectrometer (plasma viewed radially or axially) equipped for internal standardization, and capable of performing automatic background correction and interelement correction. For more information refer to the current revision of Katahdin SOP CA-632, “Operation and Maintenance of the Thermo ICAP 6500 ICP Spectrophotometer”.

4.2 Computer-controlled autosampler.

4.3 Argon gas supply – high purity.

4.4 Volumetric glassware of suitable precision and accuracy.

4.5 Automatic pipets of suitable precision and accuracy. Calibrated Eppendorf Reference pipets and Finn digital pipets are appropriate.

Refer to the appropriate instrument-specific SOP for additional required equipment.

5.0 REAGENTS

5.1 Hydrochloric acid, concentrated (HCl) – spectroscopic grade.

5.2 Nitric acid, concentrated (HNO_3) – spectroscopic grade.

5.3 Reagent water, trace metals free.

5.4 Calibration blank – reagent water containing HCl (5% v/v) and HNO_3 (5% v/v). Calibration blank solution is prepared in large volumes (up to 20 liters) and stored in a carboy. Calibration blank solution is used in establishing the analytical curve, and in all initial and continuing calibration blank determinations. This solution is also used to flush the system between standards and samples. Intermediate and working standards are prepared by diluting stock standards and intermediate standards with calibration blank solution so that all standards and blanks are acid matrix-matched to sample digestates.

5.5 Single element and multielement stock standard solutions – purchased standards prepared from high purity salts or metals, and supplied by the vendors with certificates of purity and analysis. Refer to Tables 3 and 4 for a listing of stock standards required, and to Table 8 for element concentrations in stock standards.

5.6 Intermediate standard solutions – laboratory-prepared multielement standards that are used in the subsequent preparation of working standards. Refer to Table 5 for a listing of intermediate standards required and for preparation instructions. Refer to Table 7 for element concentrations in intermediate standards.

5.7 Working standard solutions – laboratory-prepared multielement standards that are used to calibrate the instrument and to perform all necessary QC checks. Refer to Table 4 for a listing of working standards and for preparation instructions. Refer to Table 6 for element concentrations in working standards.

5.8 5 mg/L yttrium internal standard solution – add 0.5 mL 10000 mg/L yttrium stock standard to a 1000 mL volumetric flask half filled with calibration blank solution. Bring to volume with calibration blank solution.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples to be analyzed for trace metals by ICP should be collected and preserved as described in the following table.

Matrix	Container[1]	Volume / Weight	Preservation / Treatment	Holding Time
Aqueous (total)	P, G	250 mL	HNO_3 to pH < 2	6 months
Aqueous (dissolved)	P, G	250 mL	Filter, HNO_3 to pH < 2	6 months
Solid	P, G	10 g	Cool, 4°C	6 months

[1] P = polyethylene or, G = glass

7.0 PROCEDURES

7.1 Begin by following the startup and calibration instructions provided in the current revision of Katahdin SOP CA-632, "Operation and Maintenance of the Thermo ICAP 6500 ICP Spectrophotometer"

7.2 Analysis must proceed in the sequence described in Table 9 to ensure that all necessary quality control samples are analyzed at the appropriate frequencies. A minimum of two replicate integrations is required for all standards and samples. Analysis always begins with the analysis of a calibration blank solution (S0) followed by analysis of a multielement calibration standard (S1 in Table 4) to calibrate the instrument. The system is flushed with calibration blank for two minutes between each sample and standard, and each sample and standard is aspirated for one minute prior to the beginning of emission measurements.

7.3 Analysis continues with analysis of the initial calibration verification standard (ICV) and the initial calibration blank (ICB) to verify the accuracy of the calibration. Refer to Section 8 and Table 1 for additional information.

7.4 A continuing calibration verification standard (CCV) and a continuing calibration blank (CCB) must be analyzed at the beginning of the run, after every ten samples, and at the end of the run to verify the continued accuracy of the calibration. Refer to Section 8 and Table 1 for additional information.

7.5 Interference check standard solutions (ICSA and ICSAB) must be analyzed at the beginning, end, and at periodic intervals (4-6 hours, 30-40 analytical samples) throughout the sample run to verify the accuracy of the IEC factors. Refer to Section 8 and Table 1 for additional information.

7.6 A practical quantitation limit standard (PQL) must be analyzed at the beginning of each run to determine the accuracy of the calibration at the reporting limit. Refer to Section 8 and Table 1 for additional information.

7.7 All sample analytical results for a particular element that are bracketed (preceded or followed) by failing results in a QC sample (ICV, ICB, CCV, CCB, ICSA, or ICSAB) for that element must not be reported. The sample must be reanalyzed for the element in question.

7.8 All samples that exceed the linear dynamic range must be diluted and reanalyzed. This includes samples with interfering elements that exceed the calibration ranges, because accurate quantitation of interfering elements is necessary for reliable interelement correction. For example, if a sample has been submitted to the laboratory for lead analysis, and the measured aluminum concentration of that

sample exceeds the calibration range for aluminum, it must be diluted sufficiently to bring aluminum within the linear dynamic range and the lead result must be reported from that dilution analysis.

7.9 If dilutions of digested samples are performed, the measured element concentrations must be multiplied by the dilution factor prior to reporting. This is accomplished automatically by entering the dilution factor in the autosampler table prior to initiation of analysis.

7.10 All analyses are performed using yttrium as an internal standard to compensate for enhancement or depression of the analytical signal due to matrix effects. Yttrium solution is pumped at a constant rate through one channel of the peristaltic pump. Samples and standards are pumped through a second channel of the pump. The tubing carrying the internal standard is connected to the tubing carrying samples and standards downstream from the pump, and mixing of the two streams is accomplished in a mixing coil downstream from the connection, prior to nebulization. For each sample or standard, the computer that controls the spectrometer divides the detected emission signal for each element by the detected yttrium emission signal prior to quantitation, thus normalizing all emission signals to that of yttrium.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 6010 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases “qualified” data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of

the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

INITIAL DEMONSTRATION OF PERFORMANCE

8.1 Instrument detection limits (IDL) are determined quarterly for each analyte analyzed on each instrument. This determination requires seven replicate analyses of a reagent water spiked at 3-5 times the anticipated detection limit for each analyte, performed on three non-consecutive days. The standard deviation of the 21 analyses is multiplied by three to obtain the IDL. For more information on performing IDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.2 Method detection limits (MDL) are determined annually for each analyte analyzed on each instrument. This determination requires at least seven replicate digestions and analyses of a reagent water spiked at 3-5 times the anticipated MDL for each analyte. MDLs differ from IDLs in that the seven replicates are digested prior to analysis, and they may be analyzed on a single day. The standard deviation of the 7 (or more) replicate analyses is multiplied by the Student's t-value to obtain the MDL. For more information on performing MDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.3 Limits of Detection (LOD) are used when evaluating data using DoD QSM. The LOD is established by spiking a quality system matrix at 2-3 times the detection limit for a single analyte standard and 1-4 times the detection limit for a multi-analyte standard. The LOD must be verified quarterly. For more information on performing LOD determinations, refer to the current revision of Katahdin SOP QA-806.

8.4 Limits of Quantitation (LOQ) are used when evaluating data using DoD QSM. The LOQ must be above the LOD.

8.5 A Lower Limit of Quantitation Check (LLQC) sample must be prepared and analyzed annually or on an as-needed basis to confirm the laboratory's Practical Quantitation Limits (PQLs). The LLQC sample is equivalent to the PQL standard (Section 8.10) but is carried through the entire sample preparation and analysis process. Element recoveries for the LLQC sample must fall within 70% to 130% of the expected concentrations to confirm the previously established PQLs.

8.6 The upper limit of the linear dynamic range (LDR) must be established for each wavelength utilized. It must be determined from a linear calibration prepared in the normal manner using the established analytical operating procedure for the instrument. The LDR should be determined by analyzing succeedingly higher standard concentrations of the analyte until the observed analyte concentration differs by no more than 10% from the stated concentration of the standard. Determined LDRs must be documented and kept on file. The LDR which may be used for the analyses of samples should be judged by the analyst from the resulting

data. Determined sample analyte concentrations that are greater than the determined upper LDR limit must be diluted and reanalyzed. The LDRs should be verified **every six months** or whenever, in the judgment of the analyst, a change in analytical performance caused by either a change in instrument hardware or operating conditions would dictate they be redetermined.

8.7 The alkali and alkaline earth metals may have non-linear response curves due to ionization and self-absorption effects. These curves may be used for quantitation of samples if the effective range is checked and if the second order curve fit has a correlation coefficient of 0.998 or better. Third order fits are not acceptable. Non-linear response curves must be revalidated and recalculated every six months.

ANALYTICAL RUN QC SAMPLES

8.8 An Initial Calibration Verification (ICV) solution is analyzed after the initial calibration to check calibration accuracy. The ICV solution is prepared by combining compatible elements from a standard source different than that of the calibration standard and at concentrations within the linear working range of the instrument. The results of the ICV must fall within 90% to 110% of the expected values. If the ICV fails, result for the failing elements may not be reported from the run unless the ICV recovery is greater than 110% and the sample result is less than the PQL.

No results may be accepted for failing elements if DoD QSM acceptance criteria are being used.

8.9 Continuing Calibration Verification (CCV) solutions are analyzed after the initial calibration, after every ten samples, and at the end of the analytical run. The CCV solution is prepared using the same standards used for calibration at concentrations near the mid-point of the calibration curve. Results of the CCVs must fall within 90% to 110% of the expected values. If a CCV fails, results for the failing elements may not be reported from the run unless the CCV recovery is greater than 110% and the sample result is less than the PQL (less than reporting limit for DoD QSM). Also, for failing elements, all samples analyzed after the last passing CCV must be reanalyzed.

8.10 Calibration blank solution is analyzed after each ICV and CCV. A calibration blank that is analyzed after the ICV is called an Initial Calibration Blank (ICB). A calibration blank that is analyzed after a CCV is called a Continuing Calibration Blank (CCB). The absolute values of results of ICBs and CCBs must be less than the Practical Quantitation Level (PQL) for each element. If an ICB or a CCB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICB or CCB has been analyzed, with the following exception. If the result for a CCB or ICB is greater than the PQL, sample results that are less than the PQL or greater than or equal to ten times the measured CCB concentration may be reported. Also, for failing elements, all samples analyzed after the last passing CCB must be reanalyzed, with the exception noted above.

If DoD QSM acceptance criteria are being used, the absolute values of results of ICBs and CCBs must be less than the Limit of Detection (LOD). If an ICB or a CCB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICB or CCB has been analyzed.

8.11 Interference check solutions ICSA and ICSAB (refer to Section 1.1) are analyzed at the beginning of each run to verify interelement correction factors and background correction. ICSA contains interferent elements (Al, Ca, Fe, and Mg) only, at concentrations of 200 mg/L to 500 mg/L. Results for interfering elements in the ICSA must fall within 80% to 120% of the expected values. Results for unspiked elements in ICSA must fall within ± PQL if the PQL is greater than 0.01 mg/L, within ± 2xPQL if the PQL is less than or equal to 0.01 mg/L. If DoD QSM acceptance criteria are being used, the absolute value of unspiked elements must be less than the LOD. ICSAB contains interferent elements at concentrations of 200 mg/L to 500 mg/L, and analytes at concentrations of 20 mg/L or less. Results for all elements (interferents and analytes) in ICSAB must fall within 80% to 120% of the expected values. If the ICSA or ICSAB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICSA or ICSAB has been analyzed.

8.12 A Practical Quantitation Limit (PQL) Check Standard or low level continuing calibration verification (LLCCV) is analyzed at the beginning (after the ICV and ICB samples) and at the end of each run. Element concentrations in this solution are at the laboratories practical quantitation limit. Element recoveries for the PQL check Standard must fall between 70-130% of the expected values. If the PQL Check Standard fails, the results for the failing elements may not be reported from the run, unless the PQL Check Standard recovery is greater than 130% and the samples results are less than the PQL.

If DoD QSM acceptance criteria are being used, recoveries must fall between 80-120%. If the PQL Check Standard fails, the results for the failing elements may not be reported from the run.

PREPARATION BATCH QC SAMPLES

8.13 Each digestion batch of twenty or fewer samples will contain a preparation blank and a laboratory control sample. Each batch will also contain one or more of the following QC samples: laboratory control sample duplicate, sample duplicate, matrix spike sample or matrix spike sample duplicate.

8.14 A preparation blank (PBW or PBS), consisting of reagent water carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. The results of preparation blanks must be less than the Practical Quantitation Level (PQL) for each element. For DoD QSM acceptance criteria the results must be less than ½ the PQL except for common contaminants which must be less than the PQL. If a preparation blank fails, results for the failing

elements may not be reported from the digestion batch, and all associated samples must be redigested, with the following exception. If the result for a preparation blank is greater than the PQL (greater than ½ PQL for DoD), associated sample results that are less than the PQL (less than ½ PQL for DoD) or greater than or equal to ten times the measured preparation blank concentration may be reported.

8.15 A laboratory control sample (LCS), consisting of spiked reagent water or a solid reference material carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. Results for laboratory control samples must fall within 80% to 120% of the expected value, unless vendor-supplied limits (for solid reference materials) or laboratory-generated statistical limits are available. If a laboratory control sample fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested with the following exception. If the LCS fails high, sample results less than the PQL may be reported.

If DoD QSM acceptance criteria are being used, recovery for solid matrix samples must fall between 80% to 120% except for Ag, which must fall between 75% and 120%. Results may not be reported without a valid LCS and will be qualified and explained if reanalysis cannot be performed.

SAMPLE MATRIX QC SAMPLES

8.16 Matrix spiked duplicate samples are prepared at a minimum frequency of one per digestion batch. The recovery for each element in a spiked sample or spiked duplicate sample must fall within 75% to 125% of the actual value if the result for the unspiked sample is less than four times the amount of spike added. If one or both spike recoveries fail, the associated sample result must be flagged on the report of analysis. If DoD QSM acceptance criteria are being used, recoveries must be the same as stated for laboratory control samples.

The relative percent difference between sample duplicate, matrix spiked duplicate or LCS duplicate, is calculated as follows:

$$\text{RPD (\%)} = \frac{|D_1 - D_2|}{(|D_1 + D_2|)/2} \times 100$$

Where: D_1 = sample result
D_2= duplicate sample result

A control limit of 20% RPD is applied to duplicate analysis if the original sample result is greater than 50X the IDL. If the matrix spike duplicate analysis fails, the associated sample result must be flagged on the report of analysis.

8.15 A serial dilution is analyzed to check for chemical or physical interferences. If the analyte concentration of a sample is sufficiently high (minimally, 50 x IDL or 50 x

LOQ if using DoD QSM acceptance criteria), the measured concentration of a serial dilution (1:5 dilution) of the sample should agree within 90% to 110% of the original determination. The percent difference between the original sample and the serial dilution should be calculated as follows:

$$\text{Difference (\%)} = \frac{|L-S|}{S} * 100\%$$

where: L = Serial dilution result (corrected for dilution)
S = Original sample result

If the serial dilution analysis fails, a matrix interference should be suspected. The associated sample result should be flagged on the report of analysis or the sample should be reanalyzed at dilution to eliminate the interference.

For DoD QSM samples a Post-digestion Spike (PDS) addition must be performed if the serial dilution is not within acceptance criteria.

8.16 Post-digestion Spike (PDS) additions must be performed for DoD QSM samples if the serial dilution is not within acceptance criteria or if the analyte concentrations in all samples are less than 50x the LOD. The spike addition should produce a concentration that is between 10 and 100x the LOQ. The recovery of the PDS must be within 75-125%. If the PDS fails, all samples must be run by method of standard additions or appropriately flagged.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) and the limit of detection (LOD) are defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs and LODs are determined prior to sample analysis per type of instrument and filed with the Inorganic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit and Instrument Detection Limit Studies, for procedures on determining the MDL.

Refer to the current revision of Method 6010 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007, Method 6010C.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.1, 04/22/09

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-806, Method Detection Limit and Instrument Detection Limit Studies, current revision.

List of Tables & Figures

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 1

QC REQUIREMENTS

Method	QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action
USEPA 6010	Initial Calibration, minimum 1 point plus a calibration blank.	Daily prior to sample analysis.	Correlation coefficient (r) $\geq$ 0.998	Recalibrate
	Initial Calibration Verification (ICV), prepared from a second source.	Before beginning a sample run.	Recovery within $\pm$ 10% of true value.	1) Do not use results for failing elements unless the ICV > 110% and the sample < the PQL. 2) Investigate and correct 3) DoD: No samples may be run until calibration is verified
	Initial Calibration Blank (ICB)	Immediately after the ICV.	Absolute value of ICB < PQL.	1) Do not use results if $\geq$ PQL and 10x< CCB level. 2) Investigate and correct problem.
	Continuing Calibration Verification (CCV)	At beginning of run, after every 10 samples, and at end of run.	Recovery within $\pm$ 10% of true value.	1) Do not use results for failing elements unless the CCV > 110% and the sample < the PQL. 2) Investigate and correct problem.
	Continuing Calibration Blank (CCB)	After every 10 samples and at end of the run.	Absolute value of CCB < PQL.	1) Do not use results if $\geq$ PQL and < 10x CCB level. 2) Investigate and correct problem.
	Practical Quantitation Level Check Standard (PQL) (LLCCV)	At beginning and end of run.	Recovery within ± 30% of true value.	1) Do not use results for failing elements unless the ICV > 110% and the sample < the PQL. 2) Investigate and correct problem.
	Interference Check Solution A (ICSA)	At beginning and end of run.	For Al, Ca, Fe, and Mg, recovery within $\pm$ 20% of true value. For analytes not spiked, $\pm$ PQL, or, if PQL $\leq$ 0.01 mg/L, $\pm$ 2x PQL.	1) Do not use results for failing elements. 2) Investigate and correct problem.
	Interference Check Solution AB (ICSAB)	At beginning and end of run.	Recovery of each analyte within $\pm$ 20% of true value.	1) Do not use results for failing elements. 2) Investigate and correct problem.
	Preparation Blank (PBW/PBS)	One per digestion batch of 20 or fewer samples.	Less than PQL.	1) Investigate source of contamination. 2) Redigest and reanalyze all associated samples if sample concentration $\geq$ PQL and <10x the blank concentration.
	Laboratory Control Sample (LCSW/LCSS)	One per digestion batch of 20 or fewer samples.	Recovery within $\pm$ 20% of true value, unless vendor-supplied or statistical limits have been established.	1) Investigate source of problem. 2) Redigest and reanalyze all associated samples. 3) DoD: Flag specific analytes if samples cannot be reanalyzed.
	Matrix Spike Sample (S)	One per digestion batch of 20 or fewer samples.	Recovery $\pm$ 25% of true value, if sample < 4x spike added.	1) Flag results.

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 1 (cont)

QC REQUIREMENTS

Method	QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action
USEPA 6010 (cont.)	Matrix Spike Duplicate Sample (P) or sample duplicate	One per digestion batch of 20 or fewer samples.	Recovery ± 25% of true value, if sample < 4x spike added. RPD ≤20% for duplicate spikes and sample duplicates.	1) Flag results.
	Serial Dilution (L)	One per digestion batch.	If original sample result is at least 50x IDL, 5-fold dilution must agree within ± 10% of the original result. Flag result or dilute and reanalyzed sample to eliminate interference	Perform post digestion spike addition (PDS)
	Post-Digestion Spike Sample (A)	When dilution test fails or analyte concentration in all samples <50x LOD	Recovery within ± 25%.	Run associated samples by method of standard addition or flag results.
	Instrument Detection Limit (IDL) Study	Quarterly.	IDL < MDL PQL > 2-3 * the IDL	1) Repeat IDL study. 2) Raise PQL.
	Method Detection Limit (MDL) Study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		
	Lower Limit of Quantitation Check (LLQC) Sample	Digest and analyze annually or as needed to confirm PQLs	70% - 130% of true value	Re-evaluate PQLs
	Linear Range Study	Every six months	Run succeedingly higher stds until recovery not within ± 10%. Use highest passing concentration as upper limit of linear range.	Only accept data to highest passing concentration until next linear range study.
	Limit of Detection (LOD) Determination	Quarterly	LOD = 1-4X MDL	Repeat LOD Determination
	Limit of Quantification (LOQ) Determination	Quarterly	LOQ > LOD	

TABLE 2

DoD QSM VERSION 4.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria.	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification	Refer to current revision of SOP QA-806				
LOQ establishment and verification	Refer to current revision of SOP QA-806				
Instrument detection limit (IDL) study (ICP only)	At initial set-up and after significant change in instrument type, personnel, test method, or sample matrix.	IDLs shall be ≤ LOD.	NA.	NA.	Samples may not be analyzed without a valid IDL.
Linear dynamic range or high-level check standard (ICP only)	Every 6 months.	Within ± 10% of true value.	NA.	NA.	
Initial calibration (ICAL) for all analytes ICP: minimum one high standard and a calibration blank	Daily ICAL prior to sample analysis.	If more than one calibration standard is used, $r \geq 0.995$.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
Second source calibration verification (ICV)	Once after each ICAL, prior to beginning a sample run.	Value of second source for all analyte(s) within ± 10% of true value.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.

TABLE 2 (cont)

DoD QSM VERSION 4.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing calibration verification (CCV)	ICP: within ± 10% of true value; GFAA: within ± 20% of true value; CVAA: within ± 20% of true value.	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	After every 10 field samples and at the end of the analysis sequence.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Low-level calibration check standard	Daily, after one-point ICAL.	Within ± 20% of true value.	Correct problem, then reanalyze.	Flagging criteria are not appropriate.	No samples may be analyzed without a valid low-level calibration check standard. Low-level calibration check standard should be less than or equal to the reporting limit.
Method blank	One per preparatory batch.	No analytes detected > ½ RL (> RL for common lab contaminants) and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For negative blanks, absolute value < LOD.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Reprepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Calibration blank	Before beginning a sample run, after every 10 samples, and at end of the analysis sequence.	No analytes detected > LOD. For negative blanks, absolute value < LOD.	Correct problem. Re-prep and reanalyze calibration blank. All samples following the last acceptable calibration blank must be reanalyzed.	Apply B-flag to all results for specific analyte(s) in all samples associated with the blank.	
Interference check solutions (ICS)	At the beginning of an analytical run.	ICS-A: Absolute value of concentration for all non-spiked analytes < LOD (unless they are a verified trace impurity from one of the spiked analytes); ICS-AB: Within ± 20% of true value.	Terminate analysis; locate and correct problem; reanalyze ICS, reanalyze all samples.	If corrective action fails, apply Q-flag to all results for specific analyte(s) in all samples associated with the ICS.	

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 2

DoD QSM VERSION 4.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
LCS containing all analytes to be reported	One per preparatory batch.	Water: Recovery must be within + 20% of the true value Soil: Recovery must be within vendor supplied limits (varies by lot).	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix spike (MS)	One per preparatory batch per matrix	For matrix evaluation, recovery must be within + 20% of the true value.	Examine the project-specific DQOs. If the matrix spike falls outside of DoD criteria, additional quality control tests are required to evaluate matrix effects.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix.	MSD: For matrix evaluation, recovery must be within + 20% of the true value. MSD or sample duplicate: RPD ≤ 20% (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Dilution test	One per preparatory batch.	If sample concentrations > 50 x LOQ, then the five-fold dilution must agree within ± 10% of the original measurement.	Perform post-digestion spike (PDS) addition.	Flagging criteria are not appropriate.	Only applicable for samples with concentrations > 50 x LOQ.
Post-digestion spike (PDS) addition	When dilution test fails or analyte concentration in all samples < 50 x LOD.	Recovery within 75-125%.	Run all associated samples in the preparatory batch by method of standard additions (MSA) or see flagging criteria.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	Spike addition should produce a concentration of 10 – 100 x LOQ.
Method of standard additions (MSA)	When matrix interference is confirmed.	NA.	NA.	NA.	Document use of MSA in the case narrative.
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 3

SUMMARY OF METHOD MODIFICATIONS

Topic	Katahdin SOP CA-608-10	Method 6010, current revision
Apparatus/Materials		
Reagents		
Sample preservation/ handling		
Procedures		
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		
QC - Calibration Blanks	Acceptance criteria employed for 6010: ± PQL	Acceptance criteria stated in 6010: less than 10% of PQL

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 4

PREPARATION OF CALIBRATION AND QUALITY CONTROL STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
Calibration Standard (STD1 or S1)	ICP- intermediate Standard	Lab Prepared (see Table 5)	10.0
	QCS 26	High Purity Standards	1.0
Initial Calibration Verification (ICV)	QCP-CICV-3	Inorganic Ventures	0.96
	1000 mg/L Si	Inorganic Ventures	0.98
	1000 mg/L Al	High Purity Standards	0.96
	IV-28	Inorganic Ventures	0.4
	1000 mg/L Sn	Inorganic Ventures	0.04
Interference Check Sample A (ICSA)	CLPP-ICS-A	Inorganic Ventures	10.0
Interference Check Sample AB (ICSAB)	CLPP-ICS-A	Inorganic Ventures	10.0
	CLPP-ICS-B4	Inorganic Ventures	1.0
	ICSAB-INT	Lab Prepared (see Table 5)	5.0
Continuing Calibration Verification (CCV)	ICP intermediate standard	Lab Prepared (see Table 5)	5.0
	QCS 26	High Purity Standards	0.5
Practical Quantitation Limit Sample (PQL)	PQL-INT	Lab Prepared (see Table 5)	1.0

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 5

PREPARATION OF INTERMEDIATE STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
PQL-INT	1000 mg/L B,Li,Sn,Sr, W, U	H-P or IV	1.0 each
	10000 mg/L K, Na	H-P or IV	1.0 each
	1000 mg/L Ni	High Purity Standards	0.4
	1000 mg/L Co	High Purity Standards	0.3
	1000 mg/L Cu,V,Zn	High Purity Standards	0.25 each
	1000 mg/L Si	High Purity Standards	2.0
	1000 mg/L Cr,Ti,Tl,Ag	High Purity Standards	0.15 each
	1000 mg/L Cd,Se, Mo	High Purity Standards	0.1 each
	10000 mg/L Al	High Purity Standards	0.3
	1000 mg/L As,Sb	High Purity Standards	0.08 each
	1000 mg/L Ba,Be,Mn,Pb	High Purity Standards	0.05 each
	10000 mg/L Ca,Mg	High Purity Standards	0.05 each
	10000 mg/L Fe	High Purity Standards	0.1
ICSAB-INT	10000 mg/L K,Na	H-P or IV	4.0 each
	10000 mg/L B, Li, Mo,Sr,Sn,Ti, W, U	High Purity Standards	1.0 each
	1000 mg/L Si	High Purity Standards	4.0

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 6

ELEMENT CONCENTRATIONS IN WORKING STANDARDS

	CONCENTRATION IN SOLUTION, mg/L								
Element	**STD1**	**ICV**	**PQL**	**ICSA**	**ICSAB**	**CCV**	**AL_IEC**	**FE_IEC**	**MN_IEC**
Aluminum	25	10	0.3	500	500	12.5	500		
Antimony	1	0.4	0.008		0.6	0.5			
Arsenic	1	0.4	0.008		0.1	0.5			
Barium	1	0.4	0.005		0.5	0.5			
Beryllium	1	0.4	0.005		0.5	0.5			
Boron	1	0.4	0.1		0.5	0.5			
Cadmium	1	0.4	0.01		1.0	0.5			
Calcium	25	10	0.05	500	500	12.5			
Chromium	1	0.4	0.015		0.5	0.5			
Cobalt	1	0.4	0.03		0.5	0.5			
Copper	1	0.4	0.025		0.5	0.5			
Iron	25	10	0.1	200	200	12.5		200	
Lead	1	0.4	0.005		0.05	0.5			
Lithium	1	0.4	0.1		0.5	0.5			
Magnesium	25	10	0.05	500	500	12.5			
Manganese	1	0.4	0.005		0.5	0.5			10
Molybdenum	1	0.4	0.01		0.5	0.5			
Nickel	1	0.4	0.04		0.5	0.5			
Potassium	25	13.6	1		20	12.5			
Selenium	1	0.4	0.01		0.05	0.5			
Silicon	1	0.4	0.2		2	0.5			
Silver	1	0.4	0.015		0.2	0.5			
Sodium	25	10	1		20	12.5			
Strontium	1	0.4	0.1		0.5	0.5			
Thallium	1	0.4	0.015		0.1	0.5			
Tin	1	0.4	0.1		0.5	0.5			
Titanium	1	0.4	0.015		0.5	0.5			
Tungsten	1	0.4	0.1		0.5	0.5			
Uranium	1	0.4	0.1		0.5	0.5			
Vanadium	1	0.4	0.025		0.5	0.5			
Zinc	1	0.4	0.025		1.0	0.5			

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 7

ELEMENT CONCENTRATIONS IN INTERMEDIATE STANDARDS

Element	CONCENTRATION IN SOLUTION, mg/L			
	ICP Intermed STD		PQL-INT	ICSAB-INT
Aluminum	240		30	
Antimony			0.8	
Arsenic			0.8	
Barium			0.5	
Beryllium			0.5	
Boron			10	10
Cadmium			1.0	
Calcium	240		5.0	
Chromium			1.5	
Cobalt			3.0	
Copper			2.5	
Iron	240		10	
Lead			0.5	
Lithium	10		10	10
Magnesium	240		5.0	
Manganese			0.5	
Molybdenum			1.0	10
Nickel			4.0	
Potassium	150		100	400
Selenium			1.0	
Silicon	250		20	40
Silver			1.5	
Sodium	240		100	400
Strontium	10		10	10
Thallium			1.5	
Tin	10		10	10
Titanium			1.5	10
Tungsten	10		10	10
Uranium	10		10	10
Vanadium			2.5	
Zinc			2.5	

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 8

ELEMENT CONCENTRATIONS IN STOCK STANDARDS

Element	CONCENTRATION IN SOLUTION, mg/L					
	IV-28	QCS-26	2007 ICS-1	CLPP-ICS-A	CLPP-ICS-B4	QCP-CICV-3
Aluminum	100	100		5000		
Antimony	100	100			60	
Arsenic	100	100			10	500
Barium	100	100			50	
Beryllium	100	100			50	
Boron	100	100	500			
Cadmium	100	100			100	250
Calcium	100	100		5000		
Chromium	100	100			50	
Cobalt	100	100			50	
Copper	100	100			50	
Iron	100	100		2000		
Lead	100	100			5	500
Lithium	100					
Magnesium	100	100		5000		
Manganese	100	100			50	
Molybdenum	100	100	300			
Nickel	100	100			100	
Potassium	1000	1000				
Selenium	100	100			5	500
Silicon	50	50	230			
Silver	100	100			20	
Sodium	100	100				
Strontium	100					
Thallium	100	100			10	500
Tin						
Titanium	100	100	1000			
Vanadium	100	100			50	
Zinc	100	100			100	

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 9

REQUIRED ANALYTICAL SEQUENCE

Sequence Number	Standard/Sample	Purpose
1	Blank (Calibration Blank)	Initial calibration
2	S1 (Calibration Standard)	Initial calibration
3	ICV (Initial Calibration Verification)	Check calibration accuracy
4	ICB (Initial Calibration Blank)	Check calibration accuracy
5	PQL (Practical Quantitation Level Sample)	Check calibration accuracy near PQL, repeat before final CCV, CCB
6	ICSA (Interference Check Solution A)	Verify accuracy of IEC factors, repeat before final CCV, CCB
7	ICSAB (Interference Check Solution AB)	Verify accuracy of IEC factors, repeat before final CCV, CCB
8	CCV (Continuing Calibration Verification)	Check calibration stability
9	CCB (Continuing Calibration Blank)	Check calibration stability
10-19	Analyze up to 10 samples	
20	CCV (Continuing Calibration Verification)	Check calibration stability
25	CCB (Continuing Calibration Blank)	Check calibration stability
…	Continue analyzing sequences of up to 10 samples, followed by a CCV and a CCB	

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

HARDNESS BY CALCULATION

As referenced in "Standard Methods for the Examination if Water and Wastewater," Methods 2340 A & B, Hardness Introduction and Hardness by Calculation, American Public Health Association, 18th Edition, Revised 1992, total hardness is the sum of the calcium and magnesium concentrations, both expressed as calcium carbonate, in milligrams per liter.

Once the calcium and magnesium concentrations have been determined by EPA methods 6010, 6020, 200.7 or 200.8, the total hardness of an aqueous sample may be calculated as follows:

$$\text{Total Hardness, mg equivalent } CaCO_3/L = 2.497\ (Ca, mg/L) + 4.118\ (Mg, mg/L)$$

The calcium hardness of an aqueous sample may also be calculated as follows:

$$\text{Calcium Hardness, mg equivalent } CaCO_3/L = 2.497\ (Ca, mg/L)$$

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

Prepared By: George Brewer Date: 12/97

Approved By:

Group Supervisor: George Brewer Date: 01/29/01

Operations Manager: Jak C. Burton Date: 1/29/01

QA Officer: Deborah J. Nadeau Date: 1.29.01

General Manager: Dennis F. Murphy Date: 1/29/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
02 7471A	Format changes, added pollution prevention, other minor changes to sections 7, 8 and QA Table.	DN	1.29.01	1/29/01
03 7471A	Changed LeeMAN PS200 Automated Mercury Analyzer to Cetac M6100 Mercury analyzer. Revised sect. 10 to show correct reference material. Removed fig. 2 Revised sect. 4.8, 5.7 and 8.9 to reflect current practises. minor changes through out	LAD	021605	021605
04 7471A	Sect. 5.9 and 5.10 - changed preparation of intermediate mercury standards from daily to monthly. Sect. 7.8 - removed calibration blanks (LCB/CCB). They are prepared in sect. 7.6. Added weighing of boiling chips for the prep blanks. Sect. 8.3 - Removed intermediate standards	LAD	03/08	03/08
05	Revised Sections 8 and 10, and Tables 1 and 2 to update compliance from method 7471A to method 7471B.	LAD	02/09	02/09
06	Added LOD definition. Updated sections 8, 9, 10 and Table 1 for DOD QSM version 4.1 compliance.	DN	08/09	08/09

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
07	Added Table 2 with DoD QSM Version 4.1 QC Requirements	LAD	04/10	04/10

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document SOP CA-611-07, Titled Digestion and Analysis of Solid Samples for Mercury by USEPA Method 7471.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document SOP CA-611-07, Titled Digestion and Analysis of Solid Samples for Mercury by USEPA Method 7471.

.

Recipient: _____ Date: _____

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedure used by Katahdin Analytical Services, Inc. personnel for the digestion and analysis solid samples for mercury using cold vapor atomic absorption spectrophotometry.

This method is applicable to the determination of mercury in soils, sediments, bottom deposits, and sludges under USEPA Method 7471 (Test Method for Evaluating Solid Wastes, USEPA SW 846, Third Edition).

1.1 Definitions

CCB - Continuing Calibration Blank - An analyte-free solution consisting of acidified laboratory reagent grade water used to verify calibration accuracy periodically during analysis.

CCV - Continuing Calibration Verification - A midrange standard used to verify calibration accuracy periodically during analysis.

Duplicate - A second aliquot of a sample that is prepared and analyzed in the same way as the original sample in order to determine the precision of the method.

ICB - Initial Calibration Blank - An analyte-free solution consisting of acidified laboratory reagent grade water used to verify calibration accuracy.

ICV - Initial Calibration Verification - A standard made from a source independent from the calibration standards and with analyte concentrations different from those in the CCV; used to verify the accuracy of the instrument calibration.

IDL - Instrument Detection Limit - The lowest concentration of an analyte that can be determined with 95% confidence by the instrument.

LCS - Laboratory Control Sample - A standard or solid reference material that has been brought through the sample preparation process.

LOD – Limit of Detection – An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix-specific and is used for DoD QSM acceptance criteria.

PB - Preparation Blank - Laboratory reagent grade water that has been brought through the sample preparation process.

PQL - Practical Quantitation Limit - The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the IDL.

Matrix Spike - An aliquot of a sample to which a known amount of analyte has been added before digestion.

MDL - Method Detection Limit - The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of mercury by USEPA Method 7471. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in analysis of mercury by USEPA Method 7471 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to ensure that members of their group follow this SOP, to ensure that their work is properly documented, and to initiate periodic review of the associated logbooks.

1.3 Safety

Many of the samples and reagents used in cold vapor atomic absorption are toxic or corrosive. Gloves, safety glasses, lab coats, and other protective clothing should be worn whenever these materials are handled. Because of the toxic nature of mercury vapor, care must be taken to avoid its inhalation. The instrument exhaust fan must be in operation whenever the mercury analyzer is in use (the fan should never be shut off).

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address there waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

Samples, sample digestates, standards, and other reagents used in cold vapor atomic absorption may contain high concentrations of acids, mercury, and other toxic metals. They should be disposed of in a manner appropriate to the types of hazards they present. All digested mercury samples and standards and excess reagents and standards should be disposed of in the satellite waste container for corrosive wastes (labeled “Waste Stream A”) that is located in the Metals Prep lab. Further information regarding waste classification and disposal may be obtained by consulting the laboratory's Katahdin Analytical Environmental Health and Safety Manual and the Department Manager.

2.0 SUMMARY OF METHOD

The cold vapor atomic absorption technique is based on the absorption of radiation at 253.7 nm by mercury vapor. It relies on the volatility of elemental mercury at room temperature. During preparation, organic mercurials are oxidized and elemental mercury is ionized to Hg^{3+}. During instrumental analysis, mercuric ions are reduced to elemental mercury by the addition of stannous chloride. Elemental mercury is then aerated from solution and passes through a cell positioned in the path of a mercury spectrophotometer, where absorbance (peak height) is measured as a function of mercury concentration and recorded by the associated computer. The mercury vapor is then swept out of the instrument into an exhaust hood, where it is evacuated from the laboratory.

3.0 INTERFERENCES

In addition to inorganic forms of mercury, organic mercurials may be present in environmental samples. These organo-mercury compounds will not respond to the cold

vapor atomic absorption technique unless they are first broken down and converted to mercuric ions. The presence of undigested organo-mercurials in samples will result in a low bias for analytical results. Certain volatile organic materials will also non-specifically absorb radiation at the 253.7 nm analytical wavelength. The presence of such compounds may result in a high bias for analytical results. For these reasons, complete digestion using potassium permanganate is required for all environmental samples. Complete digestion is indicated by the persistence of the purple permanganate color (indicating the presence of excess permanganate) following digestion.

Samples that are high in chlorides may require additional permanganate to maintain a persistent purple color following digestion. During the oxidation step, chlorides are converted to free chlorine, which will absorb radiation at the 253.7 nm analytical wavelength. Any free chlorine thus generated will be present in the headspace of the digestion vessel following digestion. Because samples are poured into autosampler tubes prior to analysis by the mercury analyzer, any free chlorine present in the headspace of the digestion vessels is not sampled by the instrument and the analysis is free of chlorine interference.

4.0 APPARATUS AND MATERIALS

4.1 250 mL Pyrex media bottles with plastic screw caps, for use as digestion vessels.

4.2 Water bath capable of maintaining a constant temperature of 95^{o} C.

4.3 Analytical balance capable of weighing to 0.01 g.

4.4 Adjustable volume automatic pipettes - 2 to 20 uL, 10 to 100 uL, 100 to 1000 uL. Calibrated Eppendorf Reference pipets and Finn digital pipets are appropriate.

4.5 Repipetters (adjustable repeating pipetters with reservoirs) for dispensing concentrated nitric acid, concentrated sulfuric acid, and other reagents.

4.6 Spirit-filled thermometer, NIST-traceable, covering the range from 20^{o} to 110^{o} C, for monitoring the temperature of the water bath. Mercury-filled thermometers are not acceptable for use in the metals laboratory, due to the possibility of breakage and consequent contamination.

4.7 Disposable graduated polystyrene sample cups, 200 mL capacity.

4.8 CETAC M6100 Mercury Analyzer and associated peripherals and parts.

Refer to Katahdin SOP CA-629, current revision, “Operation and Maintenance of the CETAC M6100 Mercury Analyzer” for additional required materials.

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

5.0 REAGENTS

5.1 Laboratory reagent grade water – mercury-free water.

5.2 Concentrated nitric acid (HNO_3), trace metal grade

5.3 Concentrated hydrochloric acid (HCl), trace metal grade

5.4 Aqua regia: Prepare an appropriate amount immediately before use by carefully adding three volumes of concentrated HCl to one volume of concentrated HNO_3 in a heat-proof beaker or flask. Preparation of aqua regia must be performed in a fume hood.

5.5 Potassium permanganate solution, 5% w/v: Dissolve 50 g of potassium permanganate in 1 L laboratory reagent grade water. The source reagent should be labeled as suitable for use in mercury determination.

5.6 Sodium chloride – hydroxylamine hydrochloride solution: Dissolve 120 g sodium chloride and 120 g hydroxylamine hydrochloride in laboratory reagent grade water and dilute to a final volume of 1 L.

5.7 Stannous chloride solution: Add 70 mL concentrated hydrochloric acid to 500 mL of laboratory reagent grade water. Add 100 g stannous chloride and bring to a final volume of 1 L. Mix to dissolve. Reagent should be labeled as suitable for use in mercury determination.

5.8 Mercury Stock Standards: Two 10.0 mg/L mercury stock standards, obtained from separate sources, are required. The mercury concentrations of these standards must be certified by the manufacturers as traceable to NIST reference standards.

5.9 Intermediate Mercury Standard A: Appropriately dilute a mercury stock standard to obtain a solution containing 1000 ug of mercury per liter in 2% nitric acid. This intermediate standard is used to prepare calibration standards, matrix spikes, CCVs, and laboratory control samples (refer to Section 8). The identity of the stock standard currently used to prepare this intermediate standard and instructions for its dilution may be obtained by consulting the Standards Preparation Logbook maintained in the Section. Intermediate Mercury Standard A must be prepared monthly, and disposed of appropriately after use. (Note: the concentrations of all stock standards must be certified by the vendors as traceable to NIST reference materials).

5.10 Intermediate Mercury Standard B: Appropriately dilute a mercury stock standard to obtain a solution containing 1000 ug of mercury per liter in 2% nitric acid. The source of the stock standard used to prepare Intermediate Mercury Standard B must be distinct from that used to prepare Intermediate Mercury Standard A (i.e. obtained

from a separate vendor). Intermediate Mercury Standard B is used to prepare the ICV (refer to Section 8.0). The identity of the stock standard currently used to prepare this intermediate standard and instructions for its dilution may be obtained by consulting the Standards Preparation Logbook maintained in the Section. Intermediate Mercury Standard B must be prepared monthly, and disposed of appropriately after use.

5.11 Solid Reference Material: A soil with a known or empirically-established mercury concentration for use in preparing the laboratory control sample for soils. Solid reference materials should be purchased with certificates listing reference values and quality control acceptance limits. See Figure 3 for an example certificate of analysis for a solid reference material.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Soil samples to be analyzed for mercury should be collected and preserved as described in the following table.

Matrix	Container[1]	Collection Volume/ Weight	Preservation/ Treatment	Holding Time
Solid	P, G	40 g	Cool to 4°C ± 2°	28 days

[1] P = polyethylene, G = glass

7.0 PROCEDURES

BOTTLE PREPARATION

7.1 Mercury digestion bottles are reused, and must be cleaned between uses. After the previous contents of the bottles have been discarded, bottles are segregated according to whether the measured mercury concentrations of the previous contents were above the PQL (contaminated bottles) or below the PQL (uncontaminated bottles). Labels are removed from the bottles by wiping with a paper towel saturated with toluene. Both contaminated and uncontaminated bottles are then cleaned with Liquinox and water, if necessary, to remove visible grime, and rinsed thoroughly with tap water.

7.2 Uncontaminated bottles are then triple-rinsed with laboratory reagent grade water, and are ready for reuse.

7.3 Contaminated bottles are placed in a bath containing 10% HCl for at least 12 hours. After acid-leaching, these bottles are triple rinsed with laboratory reagent grade water, and are then ready for reuse.

PREPARATION OF STANDARDS, QC SAMPLES, AND BLANKS

7.4 Prior to performing the digestion, make a list of the samples that are to be digested. Enter digestion information (Katahdin Sample Numbers, Bottle IDs, QC Batch ID, preparation date, analyst initials, etc.) into the ACCESS computer database and print out a copy of the benchsheet. All necessary details of sample preparation (standards preparation information, digestion times, digestion temps, initial weights and final volumes, pertinent observations, etc.) must be recorded on this benchsheet, which will be bound in the Mercury Preparation Logbook. Refer to Figure 1 for an example page from the Mercury Preparation Logbook.

7.5 Using a silver paint marker, label clean digestion bottles with the appropriate sample numbers and standard identifications for each sample and standard to be digested.

7.6 Using calibrated adjustable pipettes, prepare calibration standards by adding 0 uL, 20 uL, 50 uL, 100 uL, 500 uL, and 1000 uL of Intermediate Mercury Standard A to separate appropriately-labeled digestion bottles. The mercury concentrations of these calibration standards will be, respectively, 0 ug/L (calibration blank), 0.2 ug/L, 0.5 ug/L, 1.0 ug/L, 5.0 ug/L, and 10.0 ug/L. The 0 ug/L, 0.2 ug/L and 0.5 ug/L standards are analyzed during analysis as the CCB, PQL standard and the CCV (refer to Section 8.0), respectively, as well as being used in the creation of the calibration curve.

7.7 Using a calibrated adjustable pipette, prepare the initial calibration verification (ICV) standard (refer to Section 8) by adding 600 uL of Intermediate Mercury Standard B to an appropriately labeled digestion bottle. The mercury concentration of the ICV will be 6.0 ug/L.

7.8 Prepare an appropriate number of preparation blanks (PBS) by adding 1.0 g of Teflon boiling chips to labeled digestion bottles.

7.9 Prepare an appropriate number of laboratory control samples (LCSS) by weighing appropriate masses of solid reference material into labeled digestion bottles. The mercury concentration of these LCSSs will depend on the solid reference material used, and the mass of each aliquot. Refer to Figure 3 for an example certificate of analysis for a solid reference material.

7.10 Matrix spikes are prepared by adding 100 uL of Intermediate Mercury Std A to each matrix spike sample. The amount of mercury added to each matrix spike increases the final digestate concentration by 1.0 ug/L.

7.11 All calibration standards, QC samples, and blanks are digested in the same manner as client samples. Refer to Sample Preparation and Digestion, Steps 7.12 through 7.16 of this SOP.

SAMPLE PREPARATION AND DIGESTION

7.12 Weigh three approximate 0.2 g portions (a total of approximately 0.6 g) of untreated sample from different parts of the sample container and place them in the bottom of a labeled digestion bottle. The purpose of using three portions is to obtain a representative sample from the sample container.

7.13 Add 5 mL of laboratory reagent grade water and 5 mL of aqua regia to each sample, standard, and QC sample. Place bottles in a water bath located in a fume hood and heat for 2 minutes at 95^{o} C. Remove the bottles from the water bath and allow them to cool in a fume hood.

7.14 Add 50 mL of laboratory reagent grade water and 15 mL of potassium permanganate solution to each digestion bottle, swirl to mix, and allow to stand for at least 15 minutes. Samples that contain large amounts of oxidizable organic matter may require additional 15 mL aliquots of potassium permanganate solution. This is indicated by the failure of the purple permanganate color to persist for the entire 15 minute waiting period. Add additional 15 mL aliquots to samples as necessary until the purple color persists for 15 minutes. If any of the samples requires these additional aliquots of permanganate, note that fact on the mercury preparation benchsheet and accordingly adjust the final volumes recorded on the benchsheet for those samples.

When a persistent purple color has been obtained for all samples, place the digestion bottles in the water bath and heat for 30 minutes at 95^{o} C. Record initial and final time and temperatures on the mercury preparation benchsheet.

7.15 Remove the bottles from water bath and allow them to cool in a fume hood. If any of the samples have become colorless during heating, add additional 15 mL aliquots of potassium permanganate solution as necessary to obtain a persistent purple color and heat for an additional 30 minutes at 95^{o} C. Record any information regarding additional permanganate aliquots on the mercury preparation benchsheet and accordingly adjust the final volumes recorded on the benchsheet for the samples affected.

7.16 Add 6 mL of sodium chloride – hydroxylamine hydrochloride solution to each digestion bottle and swirl to mix. Perform this addition in a fume hood, as chlorine gas may be evolved. This will reduce the excess permanganate, and the sample will change from purple to colorless. Add 50 mL of laboratory reagent grade water to each bottle. Wait at least 30 seconds before proceeding with analysis.

INSTRUMENTAL ANALYSIS

7.17 Digested mercury samples are analyzed using the CETAC M6100 Mercury Analyzer. Analysis is automated and is controlled by the QuickTrace software running on a dedicated PC. Detailed instructions for setting up the instrument and running samples are given Katahdin SOP CA-629, "Operation and Maintenance of the CETAC M6100 Mercury Analyzer". The following information specifically pertains to analysis of digested samples in accordance with USEPA Method 7471, and should be used in conjunction with the instructions given in Katahdin SOP CA-629.

7.18 Instrument operating conditions and quality control acceptance limits are specified in the instrument software in "templates". The template that is used to analyze digested samples in accordance with USEPA Method 7471 is named "SW846-7470-7471".

7.19 Prior to analysis, digested samples, standards, and QC samples are decanted into autosampler tubes which are placed in racks on the instrument's autosampler. The "standards" autosampler rack has 10 positions for 25 x 100 mm autosampler tubes (50 mL capacity). Tubes containing the calibration standards, the ICV, the CCV, the ICB/CCB, and the PQL standard are placed in the appropriately labeled positions in this autosampler rack.

7.20 Client samples, batch QC samples (preparation blanks and laboratory control samples), and matrix QC samples (duplicates and matrix spikes) are decanted into 17 x 100 mm autosampler tubes (15 mL capacity), which are placed in the one of the "samples" autosampler racks. The "samples" autosampler racks have 60 positions for 17 x 100 mm autosampler tubes. Instructions for filling the "samples" autosampler racks, including recording the rack position of each sample, are contained in Katahdin SOP CA-629, "Operation and Maintenance of the CETAC M6100 Mercury Analyzer".

METHOD OF STANDARD ADDITIONS

7.21 The standard addition technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample constituent that enhances or depresses the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences that cause a baseline shift. The method of standard additions shall be used for analysis of all EP extracts, on all analyses submitted as part of a delisting petition, and whenever a new sample matrix is being analyzed.

7.21.1 The simplest version of this technique is the single-addition method, in which two identical aliquots of the sample solution, each of volume V_x, are taken. To the first (labeled A) is added a known volume V_S of a standard analyte solution of concentration C_S. To the second aliquot (labeled B) is

added the same volume V_S of the solvent. The analytical signals of A and B are measured and corrected for nonanalyte signals. The unknown sample concentration C_x is calculated:

$$C_X = \frac{S_B V_S C_S}{(S_A - S_B) V_X}$$

where S_A and S_B are the analytical signals (corrected for the blank) of solutions A and B, respectively. V_s and C_s should be chosen so that S_A is roughly twice S_B on the average, avoiding excess dilution of the sample. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure.

7.21.2 Improved results can be obtained by employing a series of standard additions. To equal volumes of the sample are added a series of standard solutions containing different known quantities of the analyte, and all solutions are diluted to the same final volume. For example, addition 1 should be prepared so that the resulting concentration is approximately 50 percent of the expected absorbance from the endogenous analyte in the sample. Additions 2 and 3 should be prepared so that the concentrations are approximately 100 and 150 percent of the expected endogenous sample absorbance. The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated to zero absorbance, the point of interception of the abscissa is the endogenous concentration of the analyte in the sample. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate. An example of a plot so obtained is shown in Figure 2. A linear regression program may be used to obtain the intercept concentration.

7.21.3 For the results of this MSA technique to be valid, the following limitations must be taken into consideration:

- The apparent concentrations from the calibration curve must be linear over the concentration range of concern. For the best results, the slope of the MSA plot should be nearly the same as the slope of the standard curve. If the slope is significantly different (greater than 20%), caution should be exercised.

- The effect of the interference should not vary as the ratio of analyte concentration to sample matrix changes, and the standard addition should respond in a similar manner as the analyte.

- The determination must be free of spectral interference and corrected for nonspecific background interference.

DATA REDUCTION AND REPORTING

7.22 Results are obtained in units of ug/L in the digestate. Results that exceed the calibration range of the instrument may not be reported - the sample must be appropriately diluted and reanalyzed. Results for diluted samples must be multiplied by the dilution factor prior to reporting. If additional aliquots of potassium permanganate were added during digestion, the change in digestate final volume must be taken into account in calculating the final result. Mercury results for solid samples are reported in units of ug/g, calculated on a dry weight basis. Calculation of mercury results for solid samples is performed automatically by the Metals reporting database, as follows:

$$\text{Mercury Concentration in Solid (mg/kg dry wt.)} = \frac{(C) \times (DF) \times (FV) \times 100}{(W) \times (TS)}$$

where C = Measured digestate concentration (ug/L)
DF = Instrument dilution factor
FV = Digestate final volume (L)
W = Digested wet sample weight (g)
TS = Total Solids (%)

7.23 Results are reported down to the laboratory's practical quantitation level (PQL), unless otherwise requested. Results below the PQL should be reported as "<PQL".

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 7471 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Preparation instructions and the resulting mercury concentrations for calibration standards, QC standards, and matrix spikes are detailed in Sections 7.6 through 7.10 of this SOP. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The supervisor, Operations Manager, General Manager and/or Quality Assurance Officer may be consulted to evaluate data. Some samples

may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

INITIAL DEMONSTRATION OF PERFORMANCE

8.1 Instrument detection limits (IDL) are determined quarterly for each analyte analyzed on each instrument by each method. This determination requires seven replicate analyses of laboratory reagent grade water spiked, performed on three non-consecutive days. The standard deviation of the 21 analyses is multiplied by three to obtain the IDL. For more information on performing IDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.2 Method detection limits (MDL) are determined annually for each analyte analyzed on each instrument. This determination requires at least seven replicate digestions and analyses of laboratory reagent grade water spiked at 3-5 times the anticipated MDL for each analyte. MDLs differ from IDLs in that the replicates are digested prior to analysis, and they may be analyzed on a single day. The standard deviation of the 7 (or more) replicate analyses is multiplied by the Student's t-value to obtain the MDL. For more information on performing MDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.3 Limits of Detection (LOD) are used when evaluating data using DoD QSM. The LOD is established by spiking a quality system matrix at 2-3 times the detection limit for a single analyte standard and 1-4 times the detection limit for a multi-analyte standard. The LOD must be verified quarterly. For more information on performing LOD determinations, refer to the current revision of Katahdin SOP QA-806.

ANALYTICAL RUN QC

8.4 Instrument calibration - The instrument must be calibrated each time it is set up, and calibration standards must be digested each day that samples are digested. Calibration includes analysis of a calibration blank and five calibration standards with graduated concentrations in the appropriate range. The concentration of one of the calibration standards must be at the Practical Quantitation Level (PQL). The correlation coefficient for the calibration curve must be at least 0.995. If the

calibration curve does not pass this test, analysis must be halted, the problem corrected, and the instrument recalibrated.

8.5 An Initial Calibration Verification (ICV) solution is analyzed after the initial calibration to check calibration accuracy. The ICV solution is prepared from a standard source different than that of the calibration standard and at a concentration within the working range of the instrument. The result of the ICV must fall within 90% to 110% of the expected value. If the ICV fails, results may not be reported from the run until the problem is corrected and a passing ICV has been analyzed.

8.6 The Continuing Calibration Verification (CCV) solution is analyzed after the initial calibration, after every ten samples, and at the end of the analytical run. The CCV solution is prepared using the same standard used for calibration at a concentration near the mid-point of the calibration curve. Results of the CCVs must fall within 90% to 110% of the expected value. If a CCV fails, associated sample results may not be reported from the run until the problem is corrected and a passing CCV has been analyzed. Also, all samples analyzed after the last passing CCV must be reanalyzed. For DoD QSM acceptance criteria, samples that are below the reporting limit may be reported if the CCV reads greater than 120%.

8.7 A calibration blank is analyzed after each ICV and CCV. A calibration blank that is analyzed after the ICV is called an Initial Calibration Blank (ICB). A calibration blank that is analyzed after a CCV is called a Continuing Calibration Blank (CCB). The absolute values of results of ICBs and CCBs must be less than the Practical Quantitation Level (PQL) for each element. If samples are being run using DoD QSM criteria, the absolute values of ICBs and CCBs must be less than the Limit of Detection (LOD). If an ICB or a CCB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICB or CCB has been analyzed. Also, all samples analyzed after the last passing CCB must be reanalyzed.

8.8 A standard with a mercury concentration that is at the Practical Quantitation Limit (PQL) is analyzed at the beginning of the run to determine calibration accuracy at the reporting limit. Result of the PQL standard should fall within 70% to 130% of the expected values. No corrective action has been established at this time.

PREPARATION BATCH QC SAMPLES

8.9 Preparation blank (PBW or PBS), consisting of reagent water carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. The results of preparation blanks must be less than the Practical Quantitation Level (PQL) for each element. For DoD QSM acceptance criteria the results must be less than ½ the PQL except for common contaminants which must be less than the PQL. If a preparation blank fails, results for the failing elements may not be reported from the digestion batch, and all associated samples

must be redigested, with the following exception. If the result for a preparation blank is greater than the PQL (greater than ½ PQL for DoD), associated sample results that are less than the PQL (less than ½ PQL for DoD) or greater than or equal to ten times the measured preparation blank concentration may be reported.

8.10 A laboratory control sample (LCSS), consisting of solid reference material carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. If a laboratory control sample fails, results may not be reported from the digestion batch, and all associated samples must be redigested. The laboratory uses a reference value and statistical acceptance limits for laboratory control samples are supplied by the vendor of the solid reference material. If samples are being prepared using DoD QSM acceptance criteria, the results of the LCSS must be within 80% - 120%.

SAMPLE MATRIX QC SAMPLES

8.11 Matrix spiked duplicate samples are prepared at a minimum frequency of one per digestion batch. Matrix spike recoveries for these samples are calculated as follows:

$$\text{Recovery (\%)} = \frac{(P - S)}{A} \times 100\%$$

where:

P = Spiked sample value
S = Original sample value
A = Spike amount

The recovery for each element in a spiked sample or spiked duplicate sample must fall within 75% to 125% of the actual value if the result for the unspiked sample is less than four times the amount of spike added. If one or both spike recoveries fail, a matrix interference should be suspected and the associated sample result should be flagged on the report of analysis. If DoD QSM acceptance criteria are being used, recoveries must be the same as stated for laboratory control samples.

The relative percent difference between matrix spiked duplicate sample results is calculated as follows:

$$\text{RPD (\%)} = \frac{|D_1 - D_2|}{(|D_1 + D_2|)/2} \times 100$$

where: D_1 = Spike sample result
D_2= Spike duplicate sample result

A control limit of 20% RPD is applied to matrix spike duplicate analysis. If the matrix spike duplicate analysis fails, the associated sample result should be flagged on the report of analysis.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) and the limit of detection (LOD) are defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs and LODs are determined prior to sample analysis per type of instrument and filed with the Inorganic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revision of Method 7471 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007, Method 7471B.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.1, 04/22/09.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications.

QuickTrace M6100 Mercury Analyzer Operator Manual Version 1.0.1, CETAC Technologies

QuickTrace Mercury Analyzer Software Manual, CETAC Technologies.

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TABLE 1

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Mercury/ USEPA Method 7471B	Initial Calibration, 5 points plus a calibration blank.	Daily prior to sample analysis.	Correlation coefficient ≥ 0.995.	Correct problem and repeat calibration.
	Initial Calibration Verification (ICV), prepared from a second source.	Before beginning a sample run.	Recovery within ± 10% of true value.	Correct problem and repeat calibration.
	Initial Calibration Blank (ICB)	Before beginning a sample run.	Less than PQL.	Correct problem and repeat calibration.
	Practical Quantitation Level Standard (PQL)	Before beginning a sample run.	Recovery within ± 30% of true value.	No corrective action required at this time.
	Continuing Calibration Verification (CCV)	At beginning or run, after every 10 samples, and at end of the run	Recovery within ± 10% of true value	Repeat calibration and reanalyze all samples analyzed since the last successful CCV.
	Continuing Calibration Blank (CCB)	At beginning or run, after every 10 samples, and at end of the run	Less than PQL.	Repeat calibration and reanalyze all samples analyzed since the last successful CCB.
	Preparation Blank (PBS)	One per digestion batch of 20 or fewer samples.	Less than PQL.	1) Investigate source of contamination. 2) Redigest and reanalyze all associated samples if sample concentration ≥ PQL and < 10x the blank concentration.
	Laboratory Control Sample (LCSS)	One per digestion batch of 20 or fewer samples.	Recovery within vendor-supplied acceptance limits.	Redigest all affected samples.
	Matrix Spike Sample (S)	One per digestion batch of 20 or fewer samples.	Recovery ± 25% of true value, if sample > 4x spike value.	Flag results.
	Matrix Spike Duplicate Sample (P) or sample duplicate (D)	One per digestion batch of 20 or fewer samples.	Recovery ± 25% of true value, if sample < 4x spike added.	Flag results
	Post-Digestion Matrix Spike Sample (PDS)	When matrix spike or MSD fail	Recovery ±20% of true value	Analyze serial dilution of sample
	Serial Dilution Test (L)	One per digestion batch or when PDS fails	1:5 dilution of sample must agree within 10% with undiluted result	If MS, MSD, PDS, and serial dilution fail, quantitate sample by method of standard additions
	Instrument Detection Limit (IDL) Study	Quarterly.	IDL < PQL	1)Repeat IDL study. 2)Raise PQL.

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TABLE 1

QC REQUIREMENTS (CONTINUED)

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Mercury/ USEPA Method 7471B	Method Detection Limit (MDL) Study Annually.	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		
	Limit of Detection (LOD) determination	Quarterly.	LOD = 2-3X MDL	Repeat LOD Determination.

TABLE 2

DoD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria.	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification	(Refer to current revision of SOP QA-806)				
LOQ establishment and verification	(Refer to current revision of SOP QA-806)				
Initial calibration (ICAL) for Mercury: minimum 5 standards and a calibration blank	Daily ICAL prior to sample analysis.	If more than one calibration standard is used, $r \geq 0.995$.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
Second source calibration verification (ICV)	Once after each ICAL, prior to beginning a sample run.	Value of second source for all analyte(s) within ± 10% of true value.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Continuing calibration verification (CCV)	CVAA: within ± 20% of true value.	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	After every 10 field samples and at the end of the analysis sequence.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

TABLE 2

DoD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method blank	One per preparatory batch.	No analytes detected > ½ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. Contact Client if samples cannot be reprepped within hold time. For negative blanks, absolute value < LOD.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Reprepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result. Contact Client if samples cannot be reprepped within hold time.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Calibration blank	Before beginning a sample run, after every 10 samples, and at end of the analysis sequence.	No analytes detected > LOD. For negative blanks, absolute value < LOD.	Correct problem. Re-prep and reanalyze calibration blank. All samples following the last acceptable calibration blank must be reanalyzed.	Apply B-flag to all results for specific analyte(s) in all samples associated with the blank.	
LCS containing all analytes to be reported	One per preparatory batch.	Water: Recovery must be within ± 20% of the true value Soil: Recovery must be within vendor supplied limits (varies by lot).	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix spike (MS)	One per preparatory batch per matrix (see Box D-7).	Recovery must be within ± 20% of the true value	Examine the project-specific DQOs. If the matrix spike falls outside of DoD criteria, additional quality control tests are required to evaluate matrix effects.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix	MSD: Recovery must be within ± 20% of the true value. MSD or sample duplicate: RPD ≤ 20% (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

TABLE 2

DoD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method of standard additions (MSA)	When matrix interference is confirmed.	NA.	NA.	NA.	Document use of MSA in the case narrative.
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

TABLE 3

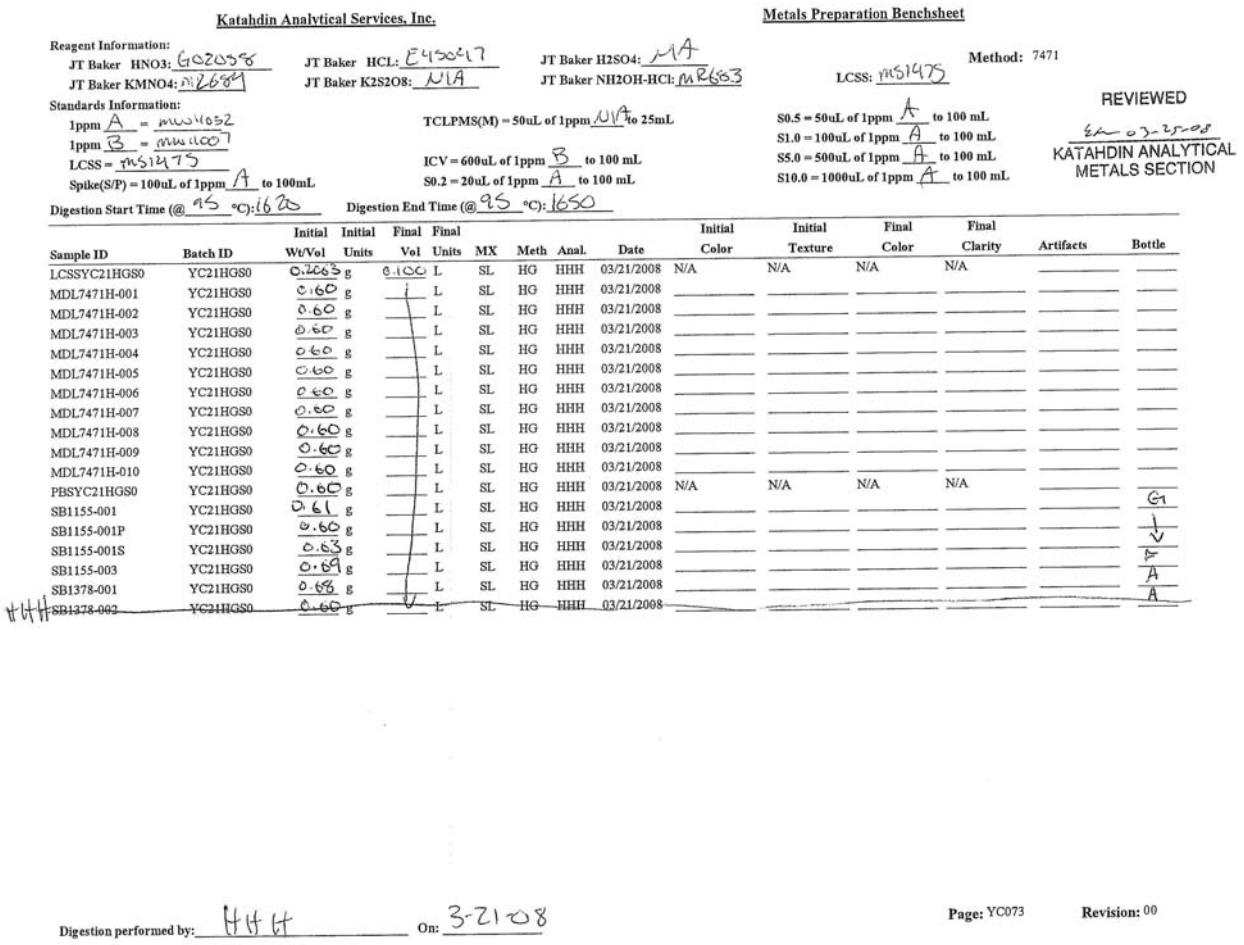
SUMMARY OF METHOD MODIFICATIONS

Topic	Katahdin SOP CA-611-07	USEPA Method 7471, current revision
Reagents	Stannous chloride dissolved in hydrochloric acid to prevent clogging of mercury analyzer, per instrument manufacturer's recommendation.	Stannous chloride dissolved/suspended in sulfuric acid.
Procedures	Sampling and gas stream switching performed automatically by mercury analyzer.	Sampling and gas stream switching performed manually by analyst.
QC – Calibration Verification	1)Known reference sample (ICV) analyzed daily. 2)Calibration verified after every 10 samples with CCV.	1)Known reference sample analyzed quarterly. 2)Calibration verified after every 20 samples.
QC - Calibration Blanks and Method Blanks	Acceptance Criterion: < PQL	Acceptance criteria: Low enough not to interfere with data quality objectives, or <10% of PQL, or <10% of regulatory limit, or <10% of lowest associated sample

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

FIGURE 1

EXAMPLE PAGE FROM MERCURY PREPARATION LOGBOOK



Katahdin Analytical Services, Inc.

Metals Preparation Benchsheet

Reagent Information:
JT Baker HNO3: G02058 JT Baker HCL: E43C17 JT Baker H2SO4: NA
JT Baker KMNO4: N26891 JT Baker K2S2O8: N/A JT Baker NH2OH-HCl: MR6563
LCSS: MS1475 Method: 7471

Standards Information:
1ppm A = MW14052
1ppm B = MW14007
LCSS = MS1475
Spike(S/P) = 100uL of 1ppm A to 100mL
TCLPMS(M) = 50uL of 1ppm N/A to 25mL
ICV = 600uL of 1ppm B to 100 mL
S0.2 = 20uL of 1ppm A to 100 mL
S0.5 = 50uL of 1ppm A to 100 mL
S1.0 = 100uL of 1ppm A to 100 mL
S5.0 = 500uL of 1ppm A to 100 mL
S10.0 = 1000uL of 1ppm A to 100 mL

REVIEWED
EA 03-25-08
KATAHDIN ANALYTICAL METALS SECTION

Digestion Start Time (@ 95 °C): 1620 Digestion End Time (@ 95 °C): 1650

Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Vol	Final Units	MX	Meth	Anal.	Date	Initial Color	Initial Texture	Final Color	Final Clarity	Artifacts	Bottle
LCSSYC21HGS0	YC21HGS0	0.2063	g	0.100	L	SL	HG	HHH	03/21/2008	N/A	N/A	N/A	N/A		
MDL7471H-001	YC21HGS0	0.60	g		L	SL	HG	HHH	03/21/2008						
MDL7471H-002	YC21HGS0	0.60	g		L	SL	HG	HHH	03/21/2008						
MDL7471H-003	YC21HGS0	0.60	g		L	SL	HG	HHH	03/21/2008						
MDL7471H-004	YC21HGS0	0.60	g		L	SL	HG	HHH	03/21/2008						
MDL7471H-005	YC21HGS0	0.60	g		L	SL	HG	HHH	03/21/2008						
MDL7471H-006	YC21HGS0	0.60	g		L	SL	HG	HHH	03/21/2008						
MDL7471H-007	YC21HGS0	0.60	g		L	SL	HG	HHH	03/21/2008						
MDL7471H-008	YC21HGS0	0.60	g		L	SL	HG	HHH	03/21/2008						
MDL7471H-009	YC21HGS0	0.60	g		L	SL	HG	HHH	03/21/2008						
MDL7471H-010	YC21HGS0	0.60	g		L	SL	HG	HHH	03/21/2008						
PBSYC21HGS0	YC21HGS0	0.60	g		L	SL	HG	HHH	03/21/2008	N/A	N/A	N/A	N/A		
SB1155-001	YC21HGS0	0.61	g		L	SL	HG	HHH	03/21/2008						G
SB1155-001P	YC21HGS0	0.60	g		L	SL	HG	HHH	03/21/2008						↓
SB1155-001S	YC21HGS0	0.63	g		L	SL	HG	HHH	03/21/2008						↓
SB1155-003	YC21HGS0	0.69	g		L	SL	HG	HHH	03/21/2008						A
SB1378-001	YC21HGS0	0.68	g		L	SL	HG	HHH	03/21/2008						A
HHH SB1378-002	YC21HGS0	0.60	g		L	SL	HG	HHH	03/21/2008						

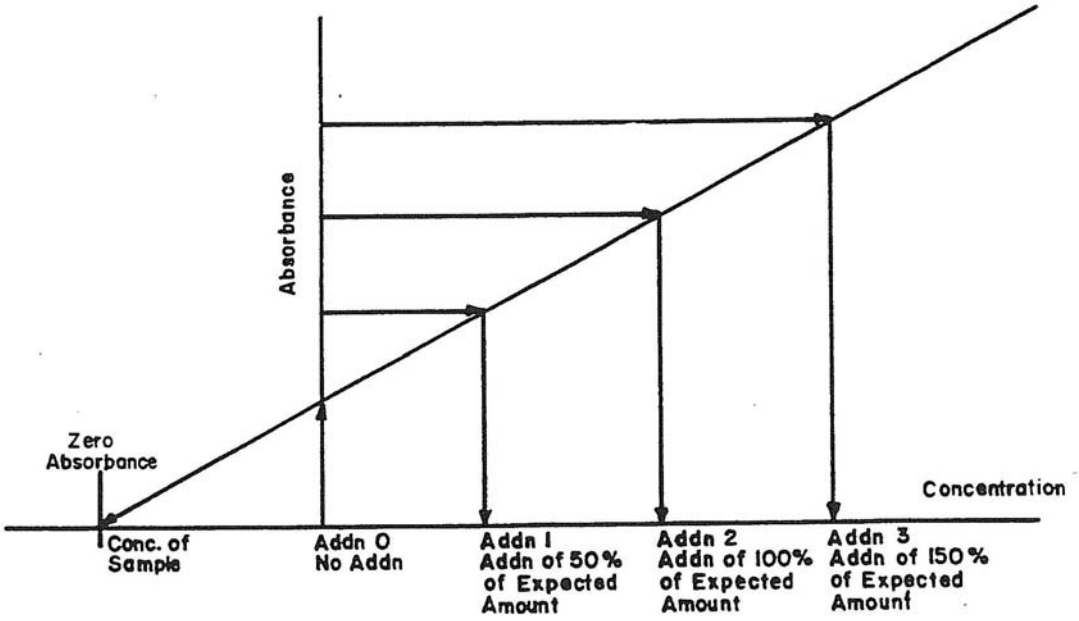
Digestion performed by: HHH On: 3-21-08

Page: YC073 Revision: 00

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

FIGURE 2

STANDARD ADDITIONS PLOT



TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

FIGURE 3

EXAMPLE CERTIFICATE OF ANALYSIS FOR A SOLID REFERENCE MATERIAL

ENVIRONMENTAL RESOURCE ASSOCIATES®
The Industry Standard™

MS1475

DataPacK™
Lot No. D051-540

Trace Metals in Soil
Catalog No. 540

Certification

Method 3050 HNO3, H2O2, HCl Parameter	Total Concentration [1] (mg/Kg)	Certified Value [2] (mg/Kg)	Performance Acceptance Limits™ [3] (mg/Kg)
aluminum	55600*	7870	4630 - 11100
antimony	160	70.5	D.L. - 149
arsenic	316	289	234 - 344
barium	869	211	174 - 247
beryllium	60.9	54.4	45.2 - 63.6
boron	129	91.3	58.8 - 124
cadmium	114	101	82.9 - 119
calcium	9750*	3680	2970 - 4390
chromium	249	224	180 - 268
cobalt	113	101	82.7 - 119
copper	94.9	88.0	73.3 - 103
iron	24400*	15700	6610 - 24900
lead	184	158	129 - 187
magnesium	3780*	2260	1760 - 2750
manganese	703	420	343 - 497
mercury	5.32	5.18	3.42 - 6.87
molybdenum	80.2	69.6	55.5 - 83.7
nickel	137	120	99.1 - 141
potassium	33000*	3000	2200 - 3800
selenium	146	130	101 - 159
silver	127	104	68.9 - 139
sodium	15600*	1080	692 - 1470
strontium	326	113	90.5 - 135
thallium	106	94.0	72.8 - 115
tin	175	149	104 - 194
titanium	3100*	284	116 - 453
vanadium	151	111	85.1 - 137
zinc	311	272	215 - 329

Method 3050 HNO3, H2O2 Parameter	Total Concentration [1] mg/Kg	Certified Value [2] mg/Kg	Performance Acceptance Limits™ [3] mg/Kg
aluminum	55600*	7380	4440 - 10300
antimony	160	75.2	D.L. - 198
arsenic	316	284	225 - 343
barium	869	217	177 - 257
beryllium	60.9	53.6	42.7 - 64.5
boron	129	89.5	58.9 - 120
cadmium	114	103	83.6 - 122
calcium	9750*	3540	2800 - 4270
chromium	249	224	172 - 275
cobalt	113	101	82.0 - 120
copper	94.9	85.5	70.4 - 100
iron	24400*	12500	5480 - 19500
lead	184	162	132 - 192
magnesium	3780*	2160	1650 - 2670
manganese	703	415	330 - 500
mercury	5.32	5.18	3.42 - 6.87
molybdenum	80.2	68.8	52.7 - 84.9
nickel	137	119	98.5 - 140
potassium	33000*	2840	2160 - 3520
selenium	146	135	104 - 166
silver	127	107	49.8 - 164
sodium	15600*	1010	709 - 1310
strontium	326	111	89.0 - 133
thallium	106	99.3	76.8 - 122
tin	175	148	70.6 - 225
titanium	3100*	283	104 - 463
vanadium	151	104	70.5 - 138
zinc	311	275	222 - 328

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-615
Revision History
Cover Page
Page 1

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

Prepared By: George Brewer Date: 01/01

Approved By:

Group Supervisor: George Brewer Date: 01/29/01

Operations Manager: Jol C. Burton Date: 1/29/01

QA Officer: Deborah J. Nadeau Date: 1·29·01

General Manager: Dennis P. Hogan Date: 1/29/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
00 7470A	NA	DH	1·29·01	1/29/01
01	Revised Sect. 4, 5 and 7 to reflect current practice. Revised Sect. 8 to reflect current QC limits. Revised sect. 10 to reflect current Applicable Documents and references. Removed figure 2. Update table 1 to reflect current QC limits. Minor changes throughout	LAD	02.16.05	02.16.05
02	Updated Fig. 1 - new prep logbook page	LAD	04/08	04/08
03	Updated Figure 1 - Example of a mercury Preparation Logbook page.	LAD	03/09	03/09
04	Added LOD definition. Updated sections 8, 9, 10 and Table 1 for DOD QSM version 4.1 compliance.	DH	08/09	08/09

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
05	Added Table 2 - DoD QSM Version 4.1 QC Requirements	LAD	04/10	04/10

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-615-05,** titled **DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-615-05,** titled **DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470.**

Recipient: _____ Date: _____

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedure used by Katahdin Analytical Services, Inc. personnel for the digestion and analysis aqueous samples for mercury using cold vapor atomic absorption spectrophotometry.

This method is applicable to the determination of mercury in groundwaters, aqueous wastes, and mobility-procedure extracts under USEPA Method 7470 (Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods, SW-846, 2nd edition, 1982 (revised 1984), 3rd edition, 1986, and Updates I, II, IIA, and III 1996, Office of Solid Waste and Emergency Response, U.S. EPA.

1.1 Definitions

CCB - Continuing Calibration Blank - An analyte-free solution consisting of acidified laboratory grade reagent water used to verify calibration accuracy periodically during analysis.

CCV - Continuing Calibration Verification - A midrange standard used to verify calibration accuracy periodically during analysis.

Duplicate - A second aliquot of a sample that is prepared and analyzed in the same way as the original sample in order to determine the precision of the method.

ICB - Initial Calibration Blank - An analyte-free solution consisting of acidified laboratory grade reagent water used to verify calibration accuracy.

ICV - Initial Calibration Verification - A standard made from a source independent from the calibration standards and with analyte concentrations different from those in the CCV; used to verify the accuracy of the instrument calibration.

IDL - Instrument Detection Limit - The lowest concentration of an analyte that can be determined with 95% confidence by the instrument.

LCS - Laboratory Control Sample - A standard or solid reference material that has been brought through the sample preparation process.

LOD – Limit of Detection – An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix-specific and is used for DoD QSM acceptance criteria.

PB - Preparation Blank - Laboratory grade reagent water that has been brought through the sample preparation process.

PQL - Practical Quantitation Limit - The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the IDL.

Matrix Spike - An aliquot of a sample to which a known amount of analyte has been added before digestion.

MDL - Method Detection Limit - The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of mercury by USEPA Method 7470. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

It is the responsibility of all Katahdin technical personnel involved in analysis of mercury by USEPA Method 7470 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to ensure that members of their group follow this SOP, that their work is properly documented, and to indicate periodic review of the associated logbooks.

1.3 Safety

Many of the samples and reagents used in cold vapor atomic absorption are toxic or corrosive. Rubber gloves, safety glasses, lab coats, and other protective clothing should be worn whenever these materials are handled. Because of the toxic nature of mercury vapor, care must be taken to avoid its inhalation. The instrument exhaust fan must be in operation whenever the mercury analyzer is in use (the fan should never be shut off).

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with

the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with the Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and follow appropriate procedures such as wearing safety glasses and gloves when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location and use of all safety equipment.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

Samples, sample digestates, standards, and other reagents used in cold vapor atomic absorption may contain high concentrations of acids, mercury, and other toxic metals. They should be disposed of in a manner appropriate to the types of hazards they present. All digested mercury samples and standards and excess reagents and standards should be disposed of in the satellite waste container for corrosive wastes (labeled “Waste Stream A”) that is located in the Metals Prep lab. Further information regarding waste classification and disposal may be obtained by consulting the laboratory's Hazardous Waste Management Plan and Safety Manual and the Department Manager.

2.0 SUMMARY OF METHOD

The cold vapor atomic absorption technique is based on the absorption of radiation at 253.7 nm by mercury vapor. It relies on the volatility of elemental mercury at room temperature. During preparation, organic mercurials are oxidized and elemental mercury is ionized to Hg^{3+}. During instrumental analysis, mercuric ions are reduced to elemental mercury by the addition of stannous chloride. Elemental mercury is then aerated from solution and passes through a cell positioned in the path of a mercury spectrophotometer, where absorbance (peak height) is measured as a function of mercury concentration and recorded by the associated computer. The mercury vapor is then swept out of the instrument into an exhaust hood, where it is evacuated from the laboratory.

3.0 INTERFERENCES

In addition to inorganic forms of mercury, organic mercurials may be present in environmental samples. These organo-mercury compounds will not respond to the cold

vapor atomic absorption technique unless they are first broken down and converted to mercuric ions. The presence of undigested organo-mercurials in samples will result in a low bias for analytical results. Certain volatile organic materials will also non-specifically absorb radiation at the 253.7 nm analytical wavelength. The presence of such compounds may result in a high bias for analytical results. For these reasons, complete digestion using potassium permanganate and potassium persulfate is required for all environmental samples. Complete digestion is indicated by the persistence of the purple permanganate color (indicating the presence of excess permanganate) following digestion.

Sea waters, brines, and industrial effluents high in chlorides may require additional permanganate to maintain a persistent purple color following digestion. During the oxidation step, chlorides are converted to free chlorine which will absorb radiation at the 253.7 nm analytical wavelength. Any free chlorine thus generated will be present in the headspace of the digestion vessel following digestion. Because samples are poured into autosampler tubes prior to analysis by the mercury analyzer, any free chlorine present in the headspace of the digestion vessels is not sampled by the instrument and the analysis is free of chlorine interference.

4.0 APPARATUS AND MATERIALS

4.1 40 mL VOA vials, for use as digestion vessels.

4.2 250 mL Pyrex media bottles with plastic screw caps, for use in digesting calibration standards.

4.3 Water bath capable of maintaining a constant temperature of 95^{o} C.

4.4 Adjustable volume automatic pipettes - 2 to 20 uL, 10 to 100 uL, 100 to 1000 uL. Calibrated Eppendorf Reference pipets and Finn digital pipets are appropriate.

4.5 Repipetters (adjustable repeating pipetters with reservoirs) for dispensing concentrated nitric acid, concentrated sulfuric acid, and other reagents

4.6 Spirit-filled thermometer, NIST-traceable, covering the range from 20^{o} to 110^{o} C, for monitoring the temperature of the water bath. Mercury-filled thermometers are not acceptable for use in the metals laboratory, due to the possibility of breakage and consequent contamination.

4.7 Disposable graduated polystyrene sample cups, 200 mL capacity

4.8 CETAC M-6100 automated mercury analyzer and associated peripherals and parts

4.9 Disposable graduated dose cups, 30 mL capacity

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

Refer to Katahdin SOP CA-629, current revision, "Operation and Maintenance of the CETAC M-6100 Automated Mercury Analyzer" for additional required materials.

5.0 REAGENTS

5.1 Laboratory grade reagent water – mercury-free water meeting the specifications of ASTM Type II water

5.2 Concentrated sulfuric acid, trace metals grade

5.3 Concentrated nitric acid, trace metals grade

5.4 Concentrated hydrochloric acid, trace metal grade

5.5 Potassium permanganate solution, 5% w/v: Dissolve 50 g of potassium permanganate in 1 L laboratory grade reagent water. The source reagent should be labeled as suitable for use in mercury determination.

5.6 Potassium persulfate solution, 5% w/v: Dissolve 50g of potassium permanganate in 1L laboratory grade reagent water. The source reagent should be labeled as suitable for use in mercury determination.

5.7 Sodium chloride – hydroxylamine hydrochloride solution: Dissolve 120 g sodium chloride and 120 g hydroxylamine hydrochloride in laboratory grade reagent water and dilute to a final volume of 1 L.

5.8 Stannous chloride solution: Add 70 mL concentrated hydrochloric acid to 500 mL of laboratory grade reagent water. Add 100 g stannous chloride and bring to a final volume of 1 L. Mix to dissolve. Reagent should be labeled as suitable for use in mercury determination.

5.9 Intermediate Mercury Standard A: Appropriately dilute a mercury stock standard to obtain a solution containing 1000 ug of mercury per liter in 2% nitric acid. This intermediate standard is used to prepare calibration standards, matrix spikes, CCVs, and laboratory control samples (refer to Section 8). The identity of the stock standard currently used to prepare this intermediate may be obtained by consulting the Standards Preparation Logbook maintained in the Section. Intermediate Mercury Standard A must be prepared fresh monthly ,and disposed of appropriately after use. (Note: the concentrations of all stock standards must be certified by the vendors as traceable to NIST reference materials).

5.10 Intermediate Mercury Standard B: Appropriately dilute a mercury stock standard to obtain a solution containing 1000 ug of mercury per liter in 2% nitric acid. The source of the stock standard used to prepare Intermediate Mercury Standard B must

be distinct from that used to prepare Intermediate Mercury Standard A (i.e. obtained from a separate vendor). Intermediate Mercury Standard B is used to prepare the ICV (refer to Section 8). The identity of the stock standard currently used to prepare this intermediate standard may be obtained by consulting the Standards Preparation Logbook maintained in the Section. Intermediate Mercury Standard B must be prepared fresh monthly, and disposed of appropriately after use.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Aqueous samples to be analyzed for mercury should be collected and preserved as described in the following table.

Matrix	Container[1]	Collection Volume/ Weight	Preservation/ Treatment	Holding Time
Aqueous (total)	P, G	250 mL	HNO_3 to pH < 2	28 days
Aqueous (dissolved)	P, G	250 mL	HNO_3 to pH < 2	28 days

[1] P = polyethylene or G = glass

7.0 PROCEDURES

BOTTLE PREPARATION

7.1 Mercury digestions are performed in two different types of vessels. Calibration standards, the Initial Calibration Verification (ICV) standard, and the Initial/Continuing Calibration Blank (ICB/CCB) are prepared in 250 mL Pyrex media bottles. Large bottles are used to provide sufficient volumes of these standards to allow for multiple reanalyses when required. Field samples, Method Blanks, and Laboratory Control Samples are digested in 40 mL VOA vials. These smaller vials provide enough digestate to allow one or two reanalyses when required, but reduce the amounts of samples consumed and waste generated.

VOA vials are reused if the samples they have contained have no measurable mercury above the PQL. After the previous contents of the vials have been discarded, these vials are segregated according to whether the measured mercury concentrations of the previous contents were above the PQL (contaminated vials) or below the PQL (uncontaminated vials). Labels are removed from the vials by wiping with a paper towel saturated with toluene. Uncontaminated vials are rinsed with laboratory grade reagent water. Contaminated vials are discarded.

The Pyrex media bottles in which standards are prepared are emptied, rinsed, and reused. Each of these bottles is permanently marked with the concentration of the standard it contains.

PREPARATION OF STANDARDS, QC SAMPLES, AND BLANKS

7.2 Prior to performing the digestion, make a list of the samples that are to be digested. Enter digestion information (Katahdin Sample Numbers, QC Batch ID, preparation date, analyst initials, etc.) into the ACCESS Metals database and print out a copy of the sample prep bench sheet. All necessary details of sample preparation (standards preparation information, digestion times, initial and final volumes, pertinent observations, etc.) must be recorded on this spreadsheet, which will be bound in the Mercury Preparation Logbook. Refer to Figure 1 for an example page from the Mercury Preparation Logbook.

7.3 Using a silver paint marker, label clean VOA vials with the appropriate sample numbers and standard identifications for each sample and standard to be digested.

7.4 Use a bottle-top dispenser to add 100 mL of laboratory grade reagent water to 6 standards digestion bottles (250 mL media bottles). Using calibrated adjustable pipettes, prepare calibration standards by adding 0 uL, 20 uL, 50 uL, 100 uL, 500 uL, and 1000 uL of Intermediate Mercury Standard A to separate appropriately-labeled media bottles containing 100 mL of laboratory grade reagent water. The mercury concentrations of these calibration standards are, respectively, 0 ug/L (calibration blank), 0.2 ug/L, 0.5 ug/L, 1.0 ug/L, 5.0 ug/L, and 10.0 ug/L. The 0.2 ug/L and 0.5 ug/L standards are analyzed after calibration as the PQL standard and the CCV (refer to Section 8.0), respectively, as well as being used in the creation of the calibration curve.

7.5 Add 100 mL of laboratory grade reagent water to the media bottle labeled “ICV”. Using a calibrated adjustable pipette, prepare the Initial Calibration Verification standard (refer to Section 8) by adding 600 uL of Intermediate Mercury Standard B to the water in this bottle, and record the bottle number in the Mercury Preparation Logbook. The mercury concentration of the ICV is 6.0 ug/L.

7.6 Prepare an appropriate number of preparation blanks (PBW) by adding 25 mL of laboratory grade reagent water to labeled vials.

7.7 Prepare an appropriate number of laboratory control samples (LCSW) by adding 125 uL of Intermediate Mercury Standard A to labeled digestion vials containing 25 mL of laboratory grade reagent water. The mercury concentration of each LCSW is 5.0 ug/L.

7.8 Matrix spikes are prepared by adding 25 uL of Intermediate Mercury Std A to 25 mL aliquots of samples. The concentration of mercury added to each matrix spike is 1.0 ug/L.

7.9 All QC samples and blanks are digested in the same manner as client samples. Refer to Sample Preparation and Digestion, sections 7.10 through 7.13 of this SOP. The volumes of reagents added to the standards prepared in the media bottles are four times those listed in sections 7.10 through 7.13.

SAMPLE PREPARATION AND DIGESTION

7.10 Using a graduated disposable dosecup, transfer 25 mL of sample, or an aliquot diluted to 25 mL, to a digestion vial. Add 1.25 mL of concentrated sulfuric acid and 0.625 mL of concentrated nitric acid, swirling to mix after each addition. Add 3.75 mL of potassium permanganate solution, swirl to mix, and allow to stand for at least 15 minutes. Samples that contain large amounts of organic substances may require additional 3.75 mL aliquots of potassium permanganate solution. This is indicated by the failure of the purple permanganate color to persist for the entire 15 minute waiting period. Add additional 3.75 mL aliquots to samples as necessary until the purple color persists for 15 minutes. If any of the samples require these additional aliquots of potassium permanganate solution, record the additional volume used for each sample on the mercury preparation benchsheet.

7.11 Add 2 mL of potassium persulfate solution to each sample. Cap the vials and place them in a preheated water bath. Monitor the temperature of the bath with a spirit thermometer throughout the digestion. The temperature of the water bath will fall below 95^{o} C upon addition of the digestion vials. After the temperature of the bath has risen back to 95^{o} C, continue heating the samples at 95^{o} C for two hours. Record initial and final digestion times and temperatures in the mercury prepareation benchsheet.

7.12 Remove bottles from the water bath and allow to cool to room temperature. If the purple permanganate color has failed to persist after digestion in any of the samples, add additional 3.75 mL aliquots of potassium permanganate solution as required to the samples, and record these additions in the mercury preparation benchsheet. Heat the samples that required additional permanganate in the water bath at 95^{o} C for an additional two hours. Remove the bottles from the water bath and allow to cool to room temperature. If the purple color fails to persist after the second heating step, consult the Department Manager for advice on how to proceed.

7.13 Add 1.5 mL of sodium chloride – hydroxylamine hydrochloride solution to each digestion vial and swirl to mix. This will reduce the excess permanganate, and the sample will change from purple to colorless. Wait at least 30 seconds before proceeding with analysis.

INSTRUMENTAL ANALYSIS

7.14 Digested mercury samples are analyzed using the CETAC M-6100 Automated Mercury Analyzer. Analysis is automated and is controlled by the QuickTrace Mercury Analyzer software running on a dedicated PC. Detailed instructions for setting up the instrument and analyzing samples are given Katahdin SOP CA-629, "Operation and Maintenance of the CETAC M-6100 Automated Mercury Analyzer".

METHOD OF STANDARD ADDITIONS

7.15 The standard addition technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample constituent that enhances or depresses the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift. The method of standard additions shall be used for analysis of all EP extracts, on all analyses submitted as part of a delisting petition, and whenever a new sample matrix is being analyzed.

7.15.1 The simplest version of this technique is the single-addition method, in which two identical aliquots of the sample solution, each of volume V_x, are taken. To the first (labeled A) is added a known volume V_S of a standard analyte solution of concentration C_S. To the second aliquot (labeled B) is added the same volume V_S of the solvent. The analytical signals of A and B are measured and corrected for nonanalyte signals. The unknown sample concentration C_x is calculated:

$$C_X = \frac{S_B V_S C_S}{(S_A - S_B)V_X}$$

where S_A and S_B are the analytical signals (corrected for the blank) of solutions A and B, respectively. V_s and C_s should be chosen so that S_A is roughly twice S_B on the average, avoiding excess dilution of the sample. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure.

7.15.2 Improved results can be obtained by employing a series of standard additions. To equal volumes of the sample are added a series of standard solutions containing different known quantities of the analyte, and all solutions are diluted to the same final volume. For example, addition 1 should be prepared so that the resulting concentration is approximately 50 percent of the expected absorbance from the endogenous analyte in the sample. Additions 2 and 3 should be prepared so that the concentrations are approximately 100 and 150 percent of the expected endogenous sample absorbance. The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known

standards plotted on the horizontal axis. When the resulting line is extrapolated to zero absorbance, the point of interception of the abscissa is the endogenous concentration of the analyte in the sample. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate. An example of a plot so obtained is shown in Figure 3. A linear regression program may be used to obtain the intercept concentration.

7.15.3 For the results of this MSA technique to be valid, the following limitations must be taken into consideration:

- The apparent concentrations from the calibration curve must be linear over the concentration range of concern. For the best results, the slope of the MSA plot should be nearly the same as the slope of the standard curve. If the slope is significantly different (greater than 20%), caution should be exercised.

- The effect of the interference should not vary as the ratio of analyte concentration to sample matrix changes, and the standard addition should respond in a similar manner as the analyte.

- The determination must be free of spectral interference and corrected for nonspecific background interference.

DATA REDUCTION AND REPORTING

7.16 Results are obtained in concentration units (ug/L) from the instrument. Electronic instrument data files are imported into the Metals ACCESS database for data reduction. Sample preparation information (initial sample volumes and final digestate volumes) are entered directly into the Metals ACCESS database to allow calculation of final results for reporting. Results are calculated as follows:

$$\text{Mercury concentration (ug/L)} = \frac{MC \times DF \times IV}{FV}$$

Where: MC = Measured mercury concentration (ug/L)
DF = Dilution factor at instrument
IV = Initial sample volume (mL)
FV = Final digestate volume (mL)

7.17 Results that exceed the calibration range of the instrument may not be reported - the sample must be appropriately diluted and reanalyzed. Results for diluted samples should be multiplied by the dilution factor prior to reporting. If additional aliquots of potassium permanganate were added during digestion, the resulting dilution must be corrected for before reporting.

7.18 Results are reported down to the laboratory's practical quantitation level (PQL), unless otherwise requested. Results below the PQL should be reported to the PQL and flagged with a "U" qualifier.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 7470 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Preparation instructions and the resulting mercury concentrations for calibration standards, QC standards, and matrix spikes are detailed in Sections 7.4 through 7.8 of this SOP. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The Department Manager, Operations Manager, General Manager and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

INITIAL DEMONSTRATION OF PERFORMANCE

8.1 Instrument detection limits (IDL) are determined quarterly for each analyte analyzed on each instrument by each method. This determination requires seven replicate analyses of a laboratory grade reagent water spiked at 3-5 times the anticipated detection limit for each analyte, performed on three non-consecutive days. The standard deviation of the 21 analyses is multiplied by three to obtain the IDL. For more information on performing IDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.2 Method detection limits (MDL) are determined annually for each analyte analyzed on each instrument. This determination requires at least seven replicate digestions

and analyses of laboratory grade reagent water spiked at 3-5 times the anticipated MDL for each analyte. MDLs differ from IDLs in that the replicates are digested prior to analysis, and they may be analyzed on a single day. The standard deviation of the 7 (or more) replicate analyses is multiplied by the Student's t-value to obtain the MDL. For more information on performing MDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.3 Limits of Detection (LOD) are used when evaluating data using DoD QSM. The LOD is established by spiking a quality system matrix at 2-3 times the detection limit for a single analyte standard and 1-4 times the detection limit for a multi-analyte standard. The LOD must be verified quarterly. For more information on performing LOD determinations, refer to the current revision of Katahdin SOP QA-806.

8.4 Instrument calibration - The instrument must be calibrated each time it is set up, and calibration standards must be digested each day that samples are digested. Calibration includes analysis of a calibration blank and five calibration standards with graduated concentrations in the appropriate range. The concentration of one of the calibration standards must be at the Practical Quantitation Level (PQL). The intermediate standards used for preparing the calibration standards are prepared at least once per month in 2% nitric acid. Because mercury may be adsorbed onto the walls of glass and plastic containers, the calibration standards must be prepared fresh daily. The correlation coefficient for the calibration curve must be at least 0.995. If the calibration curve does not pass this test, analysis must be halted, the problem corrected, and the instrument recalibrated.

8.5 An Initial Calibration Verification (ICV) solution is analyzed after the initial calibration to check calibration accuracy. The ICV solution is prepared from a standard source different than that of the calibration standard and at a concentration within the working range of the instrument. The result of the ICV must fall within 90% to 110% of the expected value. If the ICV fails, results may not be reported from the run until the problem is corrected and a passing ICV has been analyzed.

8.6 The Continuing Calibration Verification (CCV) solution is analyzed after the initial calibration, after every ten samples, and at the end of the analytical run. The CCV solution is prepared using the same standard used for calibration at a concentration near the mid-point of the calibration curve. Results of the CCVs must fall within 80% to 120% of the expected value. If a CCV fails, associated sample results may not be reported from the run until the problem is corrected and a passing CCV has been analyzed. Also, all samples analyzed after the last passing CCV must be reanalyzed.

8.7 A calibration blank is analyzed after each ICV and CCV. A calibration blank that is analyzed after the ICV is called an Initial Calibration Blank (ICB). A calibration blank that is analyzed after a CCV is called a Continuing Calibration Blank (CCB). The absolute values of results of ICBs and CCBs must be less than the Practical

Quantitation Level (PQL) for each element. If samples are being run using DoD QSM criteria, the absolute values of ICBs and CCBs must be less than the Limit of Detection (LOD). If an ICB or a CCB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICB or CCB has been analyzed. Also, all samples analyzed after the last passing CCB must be reanalyzed.

8.8 A standard with a mercury concentration that is at the Practical Quantitation Limit (PQL) is analyzed at the beginning of the run to determine calibration accuracy at the reporting limit. Result of the PQL standard should fall within 70% to 130% of the expected values. No corrective action has been established at this time.

PREPARATION BATCH QC SAMPLES

8.9 Preparation blank (PBW or PBS), consisting of reagent water carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. The results of preparation blanks must be less than the Practical Quantitation Level (PQL) for each element. For DoD QSM acceptance criteria the results must be less than ½ the PQL except for common contaminants which must be less than the PQL. If a preparation blank fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested, with the following exception. If the result for a preparation blank is greater than the PQL (greater than ½ PQL for DoD), associated sample results that are less than the PQL (less than ½ PQL for DoD) or greater than or equal to ten times the measured preparation blank concentration may be reported.

8.10 A laboratory control sample (LCSW), consisting of spiked reagent carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. Results for laboratory control samples must fall within 80% to 120% of the expected value, unless laboratory-generated statistical limits are available. If a laboratory control sample fails, results may not be reported from the digestion batch, and all associated samples must be redigested.

SAMPLE MATRIX QC SAMPLES

8.11 Matrix spiked duplicate samples are prepared at a minimum frequency of one per digestion batch. Matrix spike recoveries for these samples are calculated as follows:

$$\text{Recovery (\%)} = \frac{(P - S)}{A} \times 100\%$$

where: P = Spiked sample value
S = Original sample value
A = Spike amount

The recovery for each element in a spiked sample or spiked duplicate sample must fall within 75% to 125% of the actual value if the result for the unspiked sample is less than four times the amount of spike added. If one or both spike recoveries fail, a matrix interference should be suspected and the associated sample result should be flagged on the report of analysis. If DoD QSM acceptance criteria are being used, recoveries must be the same as stated for laboratory control samples.

The relative percent difference between matrix spiked duplicate sample results is calculated as follows:

$$RPD\ (\%) = \frac{|D_1 - D_2|}{(|D_1 + D_2|)/2} \times 100$$

where: D_1 = Spike sample result
D_2= Spike duplicate sample result

A control limit of 20% RPD is applied to matrix spike duplicate analysis. If the matrix spike duplicate analysis fails, the associated sample result should be flagged on the report of analysis.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) and the limit of detection (LOD) are defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs and LODs are determined prior to sample analysis per type of instrument and filed with the Inorganic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revision of USEPA Method 7470 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.1, 04/22/09.

Katahdin SOP CA-101, Equipment Maintenance, current revision.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications.

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The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

Test Methods for Evaluating Solid Wastes, United States Environmental Protection Agency, USEPA SW 846, Third Edition, Final Update III (9/94), Method 7470A.

QuickTrace M6100 Mercury Analyzer Operator Manual Version 1.0.1, CETAC Technologies.

QuickTrace Mercury Analyzer Software Manual, CETAC Technologies.

List of Tables and Figures

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TABLE 1

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Mercury/ USEPA 7470	Initial Calibration, 5 points plus a calibration blank.	Daily prior to sample analysis.	Correlation coefficient ≥ 0.995.	Correct problem and repeat calibration.
	Initial Calibration Verification (ICV), prepared from a second source.	Before beginning a sample run.	Recovery within ± 10% of true value.	Correct problem and repeat calibration.
	Initial Calibration Blank (ICB)	Before beginning a sample run.	Less than PQL.	Correct problem and repeat calibration.
	Practical Quantitation Level Standard (PQL)	Before beginning a sample run.	Recovery within ± 30% of true value.	No corrective action required at this time.
	Continuing Calibration Verification (CCV)	At beginning or run, after every 10 samples, and at end of the run	Recovery within ± 20% of true value	Repeat calibration and reanalyze all samples analyzed since the last successful CCV.
	Continuing Calibration Blank (CCB)	At beginning or run, after every 10 samples, and at end of the run	Less than PQL.	Repeat calibration and reanalyze all samples analyzed since the last successful CCB.
	Preparation Blank (PBW)	One per digestion batch of 20 or fewer samples.	Less than PQL.	1) Investigate source of contamination. 2) Redigest and reanalyze all associated samples if sample concentration ≥ PQL and < 10x the blank concentration.
	Laboratory Control Sample (LCSW)	One per digestion batch of 20 or fewer samples.	Recovery within ± 20% of true value.	Redigest all affected samples.
	Matrix Spike Sample (S)	One per digestion batch of 20 or fewer samples.	Recovery ±25% of true value, if sample > 4x spike value.	Flag results.
	Matrix Spike Duplicate Sample (P)	One per digestion batch of 20 or fewer samples.	1) Recovery ± 25% of true value, if sample < 4x spike added. 2) RPD ≤20% for duplicate spikes.	Flag results
	Instrument Detection Limit (IDL) Study	Quarterly.	IDL < PQL	1) Repeat IDL study. 2) Raise PQL.
	Limit of Detection (LOD) determination	Quarterly.	LOD = 2-3X MDL	Repeat LOD Determination.
	Method Detection Limit (MDL) Study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		

TABLE 2

DOD QSM VERSION 4.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria.	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification	(Refer to current revision of SOP QA-806)				
LOQ establishment and verification	(Refer to current revision of SOP QA-806)				
Initial calibration (ICAL) for mercury - minimum 5 standards and a calibration blank	Daily ICAL prior to sample analysis.	5 points plus a calibration blank, $r \geq 0.995$.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
Second source calibration verification (ICV)	Once after each ICAL, prior to beginning a sample run.	Value of second source for all analyte(s) within ± 10% of true value.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Continuing calibration verification (CCV)	within ± 20% of true value.	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	After every 10 field samples and at the end of the analysis sequence.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

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TABLE 2

DOD QSM VERSION 4.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method blank	One per preparatory batch.	No analytes detected > ½ RL (> RL for common lab contaminants) and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Reprepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result. Contact Client if samples cannot be reprepped within hold time.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Calibration blank	Before beginning a sample run, after every 10 samples, and at end of the analysis sequence.	No analytes detected > LOD.	Correct problem. Re-prep and reanalyze calibration blank. All samples following the last acceptable calibration blank must be reanalyzed.	Apply B-flag to all results for specific analyte(s) in all samples associated with the blank.	
LCS	One per preparatory batch.	Water: Recovery must be within + 20% of the true value Soil: Recovery must be within vendor supplied limits (varies by lot).	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available (see full explanation in Appendix G).	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix spike (MS)	One per preparatory batch per matrix (see Box D-7).	Recovery must be within + 20% of the true value.	Examine the project-specific DQOs. If the matrix spike falls outside of DoD criteria, additional quality control tests are required to evaluate matrix effects.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix (see Box D-7).	MSD: Recovery must be within + 20% of the true value. MSD or sample duplicate: RPD ≤ 20% (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.

TABLE 2

DOD QSM VERSION 4.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method of standard additions (MSA)	When matrix interference is confirmed.	NA.	NA.	NA.	Document use of MSA in the case narrative.
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

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TABLE 3

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-615-04	USEPA METHOD 7470
Reagents	Stannous chloride dissolved in hydrochloric acid to prevent clogging of mercury analyzer, per instrument manufacturer's recommendation.	Stannous chloride dissolved/suspended in sulfuric acid.
Procedures	1)Sampling and gas stream switching performed automatically by mercury analyzer. 2)Working Mercury standard prepared monthly in 2% nitric; calibration standards prepared fresh daily.	1)Sampling and gas stream switching performed manually by analyst. 2)Working Mercury standard prepared fresh daily and acidity maintained at 0.15% nitric.
QC – Calibration Verification	1) Known reference sample (ICV) analyzed daily. 2) Calibration verified after every 10 samples with CCV.	1) Known reference sample analyzed quarterly. 2) Calibration verified after every 20 samples.
QC - Calibration Blanks	Acceptance criteria employed for 245.1: ± PQL	Acceptance criteria stated in 245.1: ± MDL

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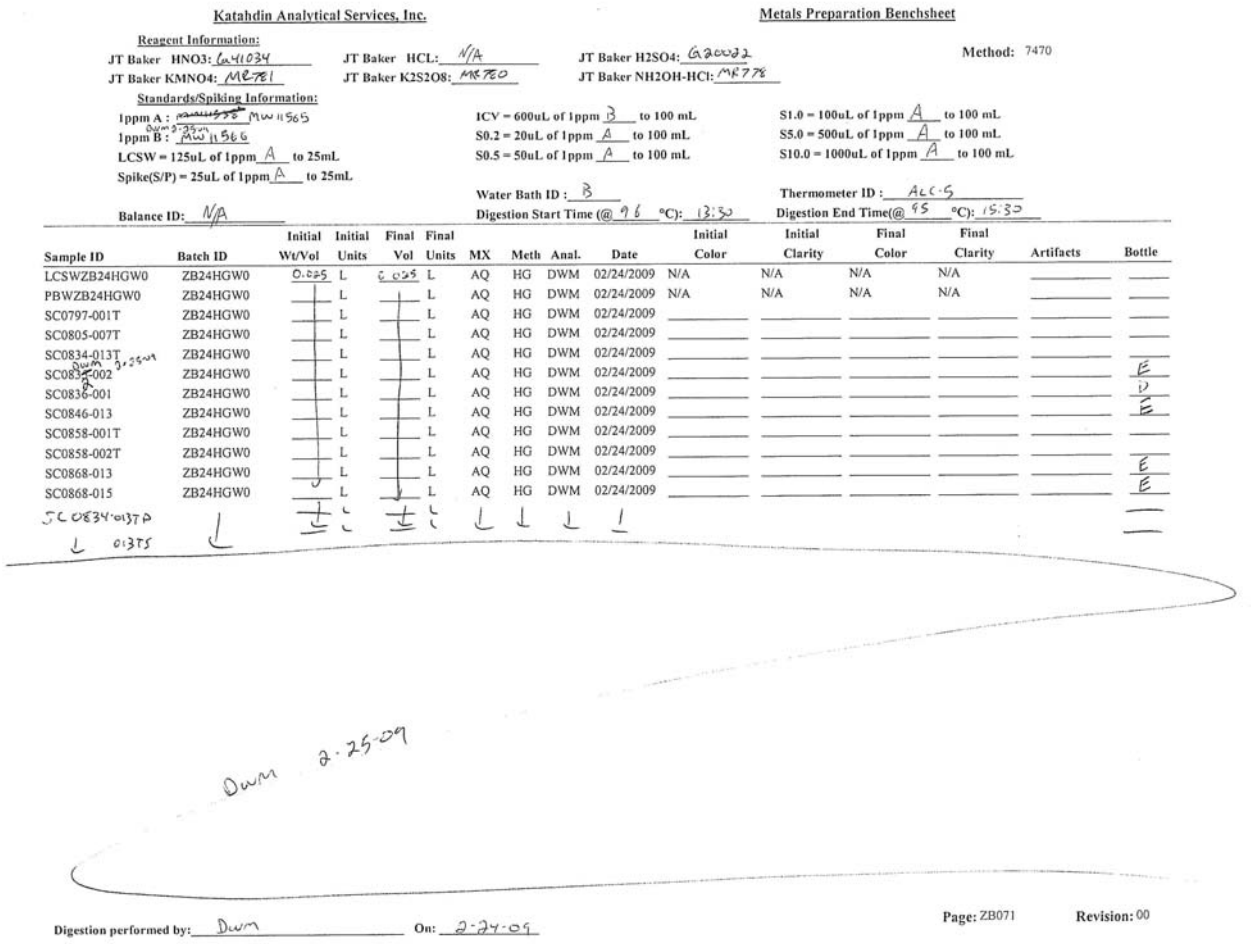
FIGURE 1

EXAMPLE PAGE FROM MERCURY PREPARATION LOGBOOK



Katahdin Analytical Services, Inc. — Metals Preparation Benchsheet

Reagent Information:
JT Baker HNO3: La41034 — JT Baker HCL: N/A — JT Baker H2SO4: G20002 — Method: 7470
JT Baker KMNO4: MR781 — JT Baker K2S2O8: MR780 — JT Baker NH2OH-HCl: MR778

Standards/Spiking Information:
1ppm A : ~~MW11538~~ MW11565
1ppm B : Dwm 2.25.09 MW11566
LCSW = 125uL of 1ppm A to 25mL
Spike(S/P) = 25uL of 1ppm A to 25mL
ICV = 600uL of 1ppm B to 100 mL
S0.2 = 20uL of 1ppm A to 100 mL
S0.5 = 50uL of 1ppm A to 100 mL
S1.0 = 100uL of 1ppm A to 100 mL
S5.0 = 500uL of 1ppm A to 100 mL
S10.0 = 1000uL of 1ppm A to 100 mL
Balance ID: N/A
Water Bath ID : B
Digestion Start Time (@ 96 °C): 13:30
Thermometer ID : ALC-5
Digestion End Time(@ 95 °C): 15:30

Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Vol	Final Units	MX	Meth	Anal.	Date	Initial Color	Initial Clarity	Final Color	Final Clarity	Artifacts	Bottle
LCSWZB24HGW0	ZB24HGW0	0.025	L	0.025	L	AQ	HG	DWM	02/24/2009	N/A	N/A	N/A	N/A		
PBWZB24HGW0	ZB24HGW0		L		L	AQ	HG	DWM	02/24/2009	N/A	N/A	N/A	N/A		
SC0797-001T	ZB24HGW0		L		L	AQ	HG	DWM	02/24/2009						
SC0805-007T	ZB24HGW0		L		L	AQ	HG	DWM	02/24/2009						
SC0834-013T	ZB24HGW0		L		L	AQ	HG	DWM	02/24/2009						
SC0834-002 (Dwm 2.25.09)	ZB24HGW0		L		L	AQ	HG	DWM	02/24/2009						E
SC0836-001	ZB24HGW0		L		L	AQ	HG	DWM	02/24/2009						V
SC0846-013	ZB24HGW0		L		L	AQ	HG	DWM	02/24/2009						W
SC0858-001T	ZB24HGW0		L		L	AQ	HG	DWM	02/24/2009						
SC0858-002T	ZB24HGW0		L		L	AQ	HG	DWM	02/24/2009						
SC0868-013	ZB24HGW0		L		L	AQ	HG	DWM	02/24/2009						E
SC0868-015	ZB24HGW0		L		L	AQ	HG	DWM	02/24/2009						E
SC0834-013TD	"		"		"	"	"	"	"						
" 0.375	"		"		"	"	"	"	"						

Dwm 2.25.09

Digestion performed by: Dwm On: 2-24-09

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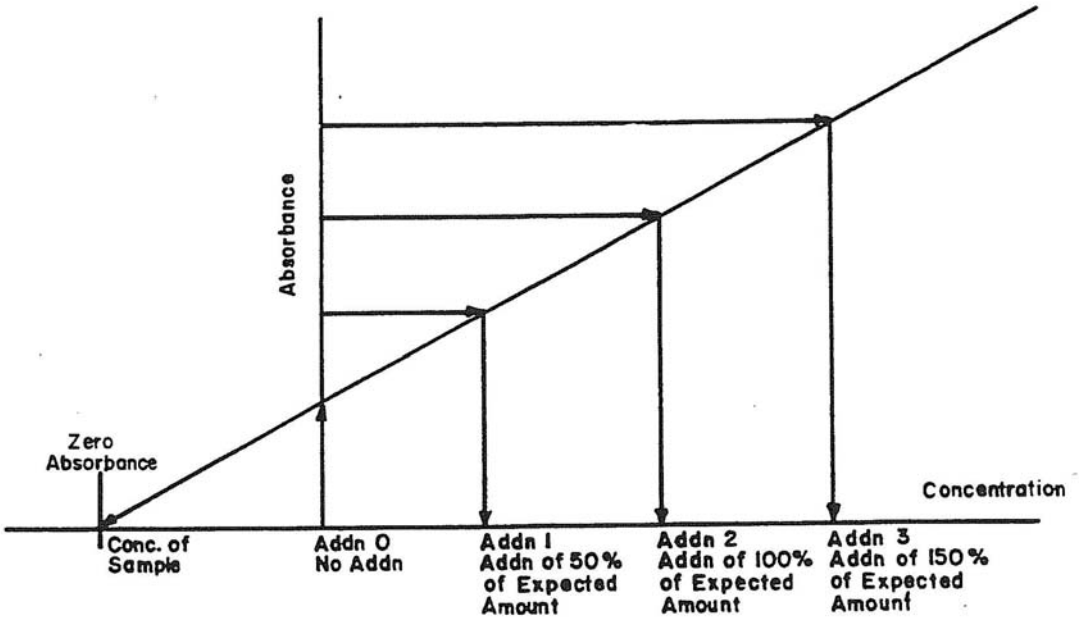
FIGURE 2

STANDARD ADDITIONS PLOT



TITLE: **SAMPLE RECEIPT AND INTERNAL CONTROL**

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Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
04	Changed cover sheet, minor changes to sections 7.1, 7.6 7.7.4, 7.10 + 7.20. Complete rewrite of sections 7.11 + 7.12 to comply with new KIMS	DN	6.6.02	6.6.02
05	Added verbal date entry to KIMS. Added reference to immediate internal COC book. Added Department Manager reference. Added section 7.7.3. Updated new incoming	DN	05.04	05.04
06	Added procedure + logbook page for checking turbidity of drinking water samples. Changed wet chem shorts board to a book (included example page). Added cwotaq procedures for food/micro. Added VOA soil freezer storage.	DN	01.26.04	01.26.04
07	Added instructions to create lettered labels. Changed sample locations to reflect new building. Removed Figures B and 10. Updated Table and Figures w/ current ones. Added wording to sect. 7.7.5 to clarify how pH measurements are taken.	LAD	02/07	02/07
08	Added summary stating sample acceptance policy. Deleted all references to radiation checks (not performed). Add IR gun usage. Reorganized section 7.0 to prioritize time sensitive tasks. Added wireless thermometer monitoring. Updated SRCR. Other minor changes.	DN	05/09	05/09

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SD-902-08**, titled **Sample Receipt and Internal Control**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SD-902-08**, titled **Sample Receipt and Internal Control**.

Recipient: _____ Date: _____

1.0 SCOPE AND APPLICATION

Katahdin Analytical Services, Inc. requires the use of specific receiving, acceptance, identification, storage, and distribution procedures for samples it accepts for analyses. These procedures assure that:

- samples are uniquely identified,
- samples are protected from loss or damage,
- essential sample characteristics are preserved,
- any alteration of samples (e.g., filtration, preservation) is documented,
- the correct samples are analyzed, and
- a record of continuous sample custody and utilization is established.

The purpose of this SOP is to describe the procedures used for the receipt and tracking of samples received by Katahdin Analytical Services, Inc. (Katahdin).

1.1 Definitions

SDG: Sample Delivery Group – A group of samples to be reported as one data package.

1.2 Responsibilities

It is the responsibility of all Katahdin staff who receive samples or handle samples in the course of analysis to follow the procedures set forth in this SOP, to document their understanding of the procedures in their training files (refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability"), and to suggest changes and revisions when appropriate. All technical staff are responsible for monitoring their immediate areas, stopping an activity when a problem is detected or suspected, initiating corrective action when needed, documenting any actions taken, and notifying the appropriate individual (e.g., Department Manager, Operations Manager, QAO). The primary responsibility for implementing real-time corrective actions and maintaining an effective QA self-inspection system resides with Katahdin staff. When problems are identified Katahdin personnel are expected to attempt to resolve situations within the scope of their technical knowledge, and to seek assistance from peers and the Department Manager as necessary.

It is the responsibility of Department Managers to oversee the adherence to Katahdin QC practices and internal documentation of laboratory activities within their area, to take corrective actions where needed and communicate problems to the Operations Manager, QAO or Vice President/President when warranted.

It is the responsibility of the Operations Manager to oversee adherence to Katahdin QA/QC practices by all laboratory groups under his/her authority, to help identify

problems and assure resolution, to facilitate corrective action where needed, and to communicate problems and concerns to the QAO and Vice President/President.

It is the responsibility of the Quality Assurance Officer (QAO) to oversee adherence to this SOP, to conduct periodic audits of each laboratory, to track corrective action reports, resolution, and documentation, and to communicate concerns and report findings to the Vice President/President. The QA Officer shall function independently from laboratory operations and be able to evaluate data objectively and perform assessments without outside influence. The QA Officer has the authority to independently halt production operations (including data reporting) if warranted by quality problems.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures and the Katahdin Environmental Health & Safety Manual and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Wastes generated during the receipt of samples must be disposed of in accordance with the Katahdin Environmental Health & Safety Manual and SOPs SD-903, "Sample Disposal" and CA-107, "The Management of Hazardous Waste as it Relates to the Disposal of Laboratory Process Waste, Reagents, Solvents and

Standards," current revisions. Expired standards are placed in the Katahdin hazardous waste storage area, and disposed of in accordance with these SOPs.

2.0 SUMMARY OF METHOD

Regulatory, program, and/or method requirements dictate the specifics of sample acceptance. These requirements include, but are not limited to, temperature upon receipt, chemical preservation, container type, sample amount, holding time considerations and complete and accurate documentation of all of these conditions, as well as sample identification. Katahdin's sample acceptance policy is to note any anomalies, discrepancies or non-compliances concerning the receipt of samples. The client is always notified with these issues to direct Katahdin on how and whether to proceed with analysis. All guidance from the client is recorded in the project phone logs and/or on the Sample Receipt Condition Report, which becomes part of the final report. Conditions or analyses performed which do not meet the necessary requirements are narrated or notated as described in the individual analytical SOPs.

3.0 INTERFERENCES

Not applicable.

4.0 APPARATUS AND MATERIALS

4.1 Thermometer – Oakton® Non-Contact Infrared Thermometer, or equivalent, capable of reading 0.1°C and digital probe style capable of reading 0.1°C (used for back-up).

4.2 Capillary tubes – 75 mm Hematocrit Tubes, disposable

4.3 Wide range pH test strips, pH 0 to 14 pH, EMD ColorpHast or equivalent.

4.4 Narrow range pH test strips, pH 0 to 2.5 pH, EMD ColorpHast or equivalent.

4.5 Narrow range pH test strips, pH 11 to 13 pH, EMD ColorpHast or equivalent.

5.0 REAGENTS

Preservatives - refer to Table 1, Sampling and Preservation Requirements, for specifics.

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Refer to Table 1, Sampling and Preservation Requirements, for specifics.

7.0 PROCEDURES

PROCEDURES FOR SAMPLE CUSTODIAN

The following procedures include all steps to be completed for satisfactory receipt and acceptance of samples at Katahdin. These steps do not necessarily have to be performed in the exact order as described. Sample deliveries occur constantly throughout the day, so the sample custodian must multi-task and move back and forth between different procedures to accomplish the most critical tasks of checking receipt temperatures and checking for "RUSH" or quick hold time parameters.

7.1 When samples (except for non-environmental food samples) are dropped off, by either a delivery service (i.e. FEDEX or UPS) or by the client, the Chain-of-Custody (COC) should be signed immediately. The client (who is delivering or that has shipped samples with a delivery service) shall sign (at the lab upon delivery or prior to shipment of samples) that they have relinquished custody to the laboratory. The laboratory shall sign and record the date and time that custody is accepted. (Refer to Figures 1-3 for a Katahdin standard COC, a Katahdin Homeowner COC, and a Katahdin Food/Microbiology COC).

7.2 Cut custody seals and open all coolers. Remove the packets containing the client Chains-of-Custody (COCs).

7.3 Using the COCs, enter the date and time of sample receipt and the client name into the next available work order/login number in the sample receipt logbook (Figure 4). Initial each entry (line) to maintain a record of the individual who assigned each group of samples a discreet lab work order/login number. Record the assigned work order numbers in the appropriate space on the client COCs. Complete the log-in entry date and time once samples are logged in as described below.

7.4 Inventory the COCs for any "RUSH" or quick hold time analyses. Notify the appropriate section managers of these analyses. List any samples for analyses that have short hold times in the "Wet Chemistry Shorts and Rushes Logbook" (Figure 5) in the wet chemistry laboratory. Be sure to list the client, number of samples and date and time of the earliest sample. GC or GC/MS personnel must be informed when ENCORES are received so that they may be scheduled for extrusion. Microbiology personnel should also be informed of any microbiology samples that arrive. Parameters that routinely require short analytical hold times are:

Coliforms	Color	pH
Nitrate/Nitrite	Dissolved Oxygen	Turbidity
Ferrous iron	Orthophosphate	Hex. Chromium
MBAS	TBOD	Free CO_2
Sulfite	ENCORE soil samples	Settleable Solids
Odor	Residual Chlorine	CBOD

7.5 Inspect the condition of custody seals, cooler, ice condition and samples received. Note any non-intact conditions on the Sample Receipt Condition Report (SRCR - Figure 6). Notify the Katahdin project manager (PM) of any discrepancies or problems with sample receipt. The PM contacts the client as necessary. If breakage of a potentially hazardous sample is discovered, close and seal the packing container with all the samples inside and move to a hood in the organic extractions area or to the smaller hood in the login area if space permits. One of the three Katahdin Emergency Response Coordinators or the Katahdin Environmental Health & Safety Manager must be notified. Disposition of the broken and other possibly contaminated samples will be determined on a case-by-case basis in accordance with the laboratory's handling procedures for hazardous waste as outlined in the Katahdin Environmental Health & Safety Manual. Generally, when a sample has broken and has mixed with any ice in the cooler, that liquid will be poured off into 2 liter plastic containers and labeled as "do not use". These containers will be disposed of as soon as the disposition of the appropriate samples has been determined through analysis.

7.6 If there is no breakage of a potentially hazardous sample:

Check cooler temperatures using the IR thermometer assigned to the sample receipt area. If a cooler temperature blank is present, aim the IR gun at the temperature blank; otherwise aim the IR gun at any sample in the cooler if no temperature blank is present. Be sure that the IR gun is within 6 inches of the bottle and not aimed at a label on the bottle. Press the trigger on the handle and be sure the red dot is visible on the bottle surface. The IR gun has been set to read in degrees celcius. If checking the temperature of a plastic bottle, set the emissivity at 0.90. If checking the temperature of a glass bottle (either amber or clear), set the emissivity at 0.85. Refer to Figure 7 for manufacturer's instructions on changing the emissivity. Record the temperature on the Sample Receipt Condition Report. Receipt temperatures should be <6 °C, without freezing. Any temperature falling outside of this range must be noted on the SRCR and reported to the appropriate Katahdin project manager.

Note: Samples received for metals analysis only do not have to meet any temperature receipt requirements.

Note: A probe type thermometer is retained as back-up in case there is a problem with the IR thermometer.

7.7 Note the condition of the ice or ice packs. If the ice has melted and the temperature is out of acceptance criteria, note this on the SRCR. For samples that are hand delivered to the laboratory immediately after collection (i.e. sample collection times are <6 hours old), the temperature blank and/or cooler temperature will most likely not meet the acceptance criteria. The samples shall be considered acceptable if there is evidence that the chilling process has begun such as arrival on ice. Note this on the SRCR. If samples (that were just collected) have not arrived on ice, note this on the SRCR, and start the cooling process as soon as possible after arrival at the laboratory.

Note: All clients must be notified when samples are received that do not meet the appropriate temperature requirements. In these cases, certain regulatory requirements may not be met and may invalidate certain data.

7.8 Inventory the samples against the chain of custody (COC). If the COC is incomplete, the sample custodian must inform the appropriate Katahdin project manager (PM). The PM may make changes to correct or complete the COC, but all changes must be initialed and dated. Changes must be noted on the SRCR. Any discrepancies between the samples and the COC must also be noted on the SRCR.

7.9 Using the Sampling and Preservation Requirements Table (Table 1) as a reference, check if samples are in proper containers and received correct pretreatment (e.g., filtration, preservation) for the analyses requested. For aqueous parameters requiring preservation, check pH by inserting a clean capillary tube into the sample and dabbing the tube on wide range pH paper. If the pH is not clearly either less than 2 or greater than 12, the appropriate narrow range pH paper must be used. NOTE: The pH of volatile organic (VOA) samples is checked and recorded by the analyst after completion of analysis and not by sample receipt personnel. The used capillary tube is discarded and a new capillary tube is used for each sample.

Additional preservative is added to samples if the pH is not in the range specified in the Sampling and Preservation Requirements Table. No more than 10% of the original sample volume should be added as preservative. If the client has noted that the sample reacts violently (i.e., foams and bubbles) upon preservation, add no more preservative to the sample. Some clients may wish to be contacted if their samples are found to be improperly preserved. Record all preservation discrepancies on the Sample Receipt Condition Report including the lot number of the preservative added. If additional preservative is added, a sticker with the type of preservative must be placed on the sample container.

Note: Preservatives are obtained from the larger containers in the bottle preparation area.

Note: If samples are received unpreserved for 200.7 or 200.8 analysis, the samples must be preserved to pH <2 with nitric acid. Samples must be held for 16 hours after preservation before sample preparation can begin.

7.10 For samples requiring filtration as pretreatment (i.e. for dissolved metals), the work order/login numbers are recorded in the filtration logbook (see Figure 8). The samples are filtered by the Metals Group.

7.10.1 A 500 mL filter flask and filter funnel are acid rinsed three times in a 10% nitric acid bath, then three times with Laboratory Reagent Grade Water.

7.10.2 A vacuum pump is attached.

7.10.3 A 0.45 micron filter is rinsed three times with 5% nitric acid and three times with Laboratory Reagent Grade Water. The rinsate is discarded.

7.10.4 A sufficient sample aliquot is filtered and preserved with concentrated nitric acid to pH <2.

7.10.5 The bottles are labeled with the work order/login number and other sample information and stored at <6 ° C until the time of digestion.

7.11 Using the Sampling and Preservation Requirements Table (Table 1) as a reference, determine if sufficient volume of sample is present for analysis. Note discrepancies on the SRCR.

7.12 For drinking water samples, enter the appropriate information (work order, date, etc.) into the Measured Turbidity and Preservation of Incoming Samples Logbook. Inform the appropriate analyst of the sample. The turbidity must be measured prior to sample preparation. If the turbidity is <1 NTU, the sample does not have to be digested prior to metals analysis. If the turbidity is >1 NTU, the sample must be digested prior to metals analysis. The sample must be preserved after the turbidity measurement is taken. Record the appropriate information in the logbook (Figure 9).

7.13 Notify the PM immediately if there are any discrepancies or problems with sample receipt. The PM will contact the client for information and resolution as necessary.

7.14 Review any additional paperwork that accompanies the sample(s) submitted for analysis along with laboratory-generated information. This includes shipping forms, letters, chain-of-custody forms, sample labels, Incoming Sample Information Sheets (ISIS), quotes, memos, etc. These forms may provide details on specific client requests. The ISIS will provide information on specifics for log-in. Refer to Figure 10 for an example.

7.15 Resolve any questions or concerns raised by steps 7.1-7.14 by consulting the correspondence files or client services personnel or communicating directly with the

client. Note in the notes section of the SRCR any deviations from normal sample handling or analytical procedures (e.g., client requests analysis although hold-time expired).

7.16 When non-environmental food samples are delivered to the laboratory, they are taken immediately to the food/microbiology laboratory and stored in the refrigerators there. A copy of the Chain-of-Custody is left with the analysts. The original paperwork is forwarded to sample log in where the job is logged into the KIMS system.

7.17 The following information is documented via the Katahdin Information Management System (KIMS) and a work order/login COC report (Figure 11) is generated for the samples received:

7.17.1 Log onto KIMS by entering employee ID under "Username", employee specific password under "Password" and KIMS under "Database".

7.17.2 Once logged onto KIMS select "Sample Management" and then "Login".

7.17.3 Select "New" and the next available Login ID number will automatically be entered. Select "OK" and the Sample Definition screen will open.

Note: If a Work Order number has already been opened, select "change" and type in the appropriate number to access the information.

7.17.4 In the Sample Definition Screen, enter the following information.

Client ID -	Enter client sample description.
ReceiveDate -	Enter in date that samples were received in the lab in the format YY-Month-DD.
CollectDate -	Enter in date that samples were collected in the format YY-Month-DDTIME.
TAT -	Enter TAT for hardcopy report.
DueDate -	Due date will automatically be calculated based on calendar days.
VerbalDate -	Manually type in verbal due date.
QuoteRef -	Enter quote number if applicable.
Project -	Enter project number if applicable.
Account -	Enter client specific account number.

Account Name - Account name will automatically be entered.

Collected By - Enter name/initials of sampler listed on COC. If unknown, enter "Client".

Locator - May be used for client ID information when requested by the project manager.

Site - Enter project site name.

Description - May be used for long client Ids when requested by the project manager.

Discount - No entry-not currently used.

Priority - No entry-not currently used.

Fact. - No entry-not currently used.

Expected - No entry-not currently used.

Comments - Enter MS/MSD, verbal due date and any sample irregularities if applicable.

OrderDate - Current date is automatically entered.

Matrix - Enter sample matrix code where

AQ = Aqueous	SLD = Food Solid
SL = Solid, Soil, Sludge	AR = Air
FP = Free Product	SWAB = Swab
WP = Wipe	SAL = Saline
NOAQ = NonAqueous	TIS = Tissue
DW = Drinking Water	

Product Code - Enter analysis code per test requested on COC.

Type - Product code type will automatically be entered where
S = Stand alone
P = Parent
C = Children

Fact. - No entry-default is 1.

Price - This is left as is by sample log-in. During project management review of the work order, the prices are entered based on quotes or standard prices.

Cost - No entry needed.

Lev - No entry needed.

Type - Container type will automatically be entered.

Bot - Enter number of containers per test for printing of labels.

Login Info - Parameter Data Screen will open. Enter following information

KAS Proj. Manager-	Initials of Katahdin person overseeing the project.
Client PO#-	Client purchase order.
Project-	Project name.
Cooler Temperature-	Temperature blanks or cooler temps.
Delivery Services-	Method of delivery to the lab.
QC Level-	QC Level of report and regulatory agency (ie., IV NFESC).
SDG ID-	Sample Delivery Group ID if applicable.
SDG Status-	Begin, Continue or End.
Analysis Instructions-	PM will enter special instructions regarding project.
Report Instructions-	PM will enter special instructions regarding project.
Regulatory List-	Used for federal programs.
EDD Format-	Specific KAS EDD format.

Select "SAVE" and then "CANCEL".

Addresses - Select "Addresses" and the Address Links screen will open. The billing address is the default address of the account. Enter the client account code under "Project/Account" and select the report to contact under "Address Type". Select the appropriate boxes for report, report CC and invoice CC. Select "SAVE" and then "CLOSE".

Create Containers - Select "Create Containers". Letters will be assigned to each sample number. Select "OK" until letters have been assigned to each sample number. To manually assign letters, Select "Enter Container IDs" and "OK". Enter sample numbers including letters and select "OK", "Close", "Yes" to save changes, "Cancel" and "Cancel".

7.17.5 To print the login report, select "Reports", "Login" and "Login COC". Enter login number under "Login Number". Select "OK", "Run Report" and then "Print".

7.18 To print labels unique to each bottle, select "Reports", "Login" and "Labels". Enter login number under "Login/Prelogin", select "Background (IDXL)" and select F9 on keyboard under "Select Sample Label". Select "OK" and then "Print". After labels print out select "Cancel".

7.19 Affix permanent sample number labels to sample containers, assuring that sample IDs on labels correspond to sample bottle IDs. Do not obscure client ID on the bottles.

7.20 Place samples in their designated storage locations and log them in, noting initials, date and time, work order/login and sample numbers, and storage location on the internal laboratory chain of custody form (Figure 12). Place form in the appropriate binder in the log in area. Non-environmental food samples do not get an internal COC and are taken immediately to the food/microbiology lab for storage.

Storage location of the samples is determined by type of sample and/or type of analysis, as outlined below. Most samples are stored in the walk-in cooler, which is organized by test type and work order/login number.

Specific storage locations are described below.

7.20.1 Aqueous samples for wet chemistry (except hardness, see 7.19.4 below) - left aisle, both sides, as you enter walk-in cooler. TOC vials are to be stored in the trays designated for TOC samples.

7.20.2 Aqueous samples for organic extractions – right aisle, left side, as you enter walk-in cooler.

7.20.3 Non-aqueous samples (all analyses except volatile organics) - to the right and towards the back as you enter walk-in cooler. Non-aqueous samples for volatile organics are stored in "VOA Refrigerator 2" located in the Volatiles Laboratory.

7.20.4 Aqueous samples for metals and/or hardness analyses – right aisle, right side towards the front as you enter walk-in cooler.

7.20.5 Samples (aqueous and solid) for volatile organics analyses (VOA) – All aqueous samples and soil samples in VOA vials (preserved with methanol or sodium bisulfate) are stored in "VOA Refrigerator 1" in the Volatiles Laboratory. VOA soils in jars or ENCORE samplers are stored in "VOA Refrigerator 2" in the Volatiles Laboratory. VOA samples known or suspected to be hazardous (such that cross-contamination of other samples might occur) are placed in a "paint can" and stored in the walk-in.

7.20.6 Soil samples for volatile organics analyses (VOA) that are unpreserved or preserved with Laboratory Reagent Grade Water are stored in "VOA Freezer 1" in the volatiles laboratory.

7.21 Sample Receipt gives the Work order/login COC report and confirmation of the job, as logged-in, to the appropriate Katahdin project manager. All chain-of-custody and other receipt documentation must accompany the job. The project manager reviews the job for accuracy and completeness. Any unresolved issues should be resolved at this time. Any project or program specific forms should be included with the paperwork at this time. These forms may include CLP forms or state-specific forms. The project manager then dispatches the work order/login to the individual department worklists. The dispatched work order/login package is then filed in Data Management where the complete package will eventually be compiled.

7.22 The temperature of all sample storage refrigerators and freezers is recorded daily by assigned individuals. Notebooks containing a record of each refrigerator and freezer temperature history are used for this purpose and are maintained by the assigned individuals. Temperatures above or below the acceptance range are to be brought to the attention of a Department Manager, Operations Manager, or Quality Assurance Officer. Such an occurrence and the actions taken to correct it must be noted in the comments column of the temperature recording notebook next to the temperature measurement. (See Figure 13).

Additionally, temperatures of storage units are monitored continuously by wireless thermometers. A temperature is recorded electronically every 10 minutes. The QAO can generate a specified report as needed, including every reading or maximum/minimum temperatures for a given timeframe. These monitoring devices ensure continual compliance seven days per week. The data can be used to check for problems.

PROCEDURES FOR CHEMISTS

7.23 When removing a sample from its storage location, record on the laboratory internal chain-of-custody (from the appropriate department) the sample number, date and

time it was removed, chemist who removed it, and the analysis to be conducted or reason for removal.

7.24 If the samples have not been logged in yet and they need to be pulled in order to analyze short holding time parameters, the analyst taking the sample must use the designated logbook (Immediate Internal COC – Figure 14) to sign the samples out. Many circumstances lead to analysts having to pull samples before they are logged into the KIMS system. It is everyone's responsibility to ensure that all samples can be accounted for at all times. Failure to do so can create confusion and bottle necks for others trying to access the samples. Samples that are pulled before log-in must be returned to the designated bin in the sample receipt area.

7.25 If a sample is not consumed by an analysis, return the remaining sample to its assigned storage location and enter the date and time returned on the laboratory internal chain-of-custody record.

7.26 If analysis consumes the entire sample, indicate this on the laboratory internal chain-of-custody record.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Each thermometer used to monitor sample storage or cooler temperatures must be calibrated annually against a NIST traceable thermometer. The QAO is responsible for ensuring that the thermometer(s) are scheduled for annual calibration and for maintaining the calibration records. All other procedures and documentation listed in this SOP must be followed at all times.

9.0 METHOD PERFORMANCE

Not applicable.

10.0 APPLICABLE DOCUMENTS/REFERENCES

"Handbook for Analytical Quality Control in Water and Wastewater Laboratories," U.S. EPA EMSL Office of Research and Development, March 1979.

Code of Federal Regulations 40, Parts 136 and 141.

"Test Methods for Evaluating Solid Waste: Physical/Chemical Methods," SW-846 Chapters 1 & 2, USEPA, Third Edition, including Updates I, II, IIA, and IIB, III June, 1997.

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

Katahdin Analytical Services, Inc., Environmental Health & Safety Manual, current revision.

LIST OF TABLES & FIGURES

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

TABLE 1

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER – AQUEOUS MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
GENERAL CHEMICAL ANALYSES					
Acidity	305.1	100 mL	P,G	1,2	14 days
Alkalinity-Manual Titrimetric	310.1	100 mL	P,G	1,2	14 days
Ammonia-Nitrogen with distill-Auto. Phenate	350.1	1 L	P,G	1,3	28 days
Ammonia-Nitrogen-Automated Phenate	350.1, 350.2	250 mL	P,G	1,3	28 days
Anions (Cl, Br, SO4, NO2, NO3)	300.0	250 mL	P, G	1	48hr/28days
Bicarbonate, Carbonate (see pH & alkalinity)	calc.				
Biochemical Oxygen Demand-Carbonaceous	405.1	1 L	P,G	1	48 hours
Biochemical Oxygen Demand-Total	405.1	1 L	P,G	1	48 hours
Bromide	320.1	500 mL	P,G	1	28 days
Chemical Oxygen Demand-Manual Colorimetric	410.4	100 mL	P,G	1,3	28 days
Chloride-Automated Ferricyanide	325.2	100 mL	P,G	1	28 days
Chlorine, Residual	SM4500-Cl G	100 mL	P,G	1,9	ASAP
Chromium, Hexavalent	SM3500Cr D / SW7196	200 mL	P,G	1,9	24 hours
Color, Apparent	110.2	100 mL	P,G	1,2	48 hours
Cyanide, Amenable-Spectrophotometric	335.1	250 mL	P,G	1,5	14 days
Cyanide, Total-Spectrophotometric	SM4500CN C 335.3, 335.4	250 mL	P,G	1,5	14 days
Dissolved Oxygen(Lab)-Membrane Electrode	360.1	500 mL	G	1	ASAP
Ferrous Iron - Colorimetric	SM3500-Fe D	250mL	P	1	24 hrs
Fluoride with distillation, Potentiometric ISE	SM4500F C/340.2	500 mL	P only	1	28 days
Fluoride, Potentiometric ISE	340.2	200 mL	P only	1	28 days
Free CO_2	SM4500-CO_2 C	250mL	P	1	24 hrs.
Hardness, Total-Manual Titrimetric	130.2,SM2340C	250 mL	P,G	4	6 months
MBAS, Extraction-Colorimetric	SM5540C	1 L	P,G	1	48 hours
Nitrate+Nitrite-Automated Cadmium Reduction	353.2	100 mL	P,G	1,3	28 days
Nitrate-Automated Cadmium Red./Diazotization	353.2	100 mL	P,G	1	48 hours
Nitrite-Automated Diazotization	353.2	100 mL	P,G	1	48 hours
Oil & Grease-Total Recoverable, Gravimetric N-Hexane extractable material N-Hexane extractable material w/ silica gel cleanup	1664	(2) 1 L	glass only	1,11	28 days
pH (Laboratory)	150.1	100 mL	P,G	1,2	24 hours
Phenolics, Total Recoverable-Manual 4AAP	420.1	1000 mL	glass only	1,3	28 days
Phosphate, Ortho- Ascorbic Acid	365.2	100 mL	P,G	1	48 hours
Phosphate,Total	365.4	100 mL	P,G	1,3	28 days
Solids-Filterable Residue (TDS),Gravimetric180	160.1	250 mL	P,G	1	7 days
Solids-Nonfilterable Residue (TSS)	160.2	500 mL	P,G	1	7 days
Solids-Settleable Solids (SS)	160.5	1 L	P,G	1	48 hours

TABLE 1 (cont.)

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER – AQUEOUS MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
GENERAL CHEMICAL ANALYSES					
Solids-Total Solids	160.3	250 mL	P,G	1	7 days
Solids-Total Volatile (TVS)	160.4	250mL	P,G	1	7 days
Solids-Volatile Filterable Residue (VDS)	160.1/160.4	250 mL	P,G	1	7 days
Solids-Volatile Nonfilterable Residue (VSS)	SM 2540 F	500 mL	P,G	1	7 days
Specific Conductance-Wheatstone Bridge	120.1	100 mL	P,G	1,2	28 days
Sulfate-Turbidimetric	375.4	100 mL	P,G	1	28 days
Sulfide-Iodometric	376.1	500 mL	P,G	1,7	7 days
Sulfite-Titrimetric	377.1	500 mL	P,G	1,9	ASAP
Tannin/Lignin-Colorimetric	SM 5550 B	100 mL	P,G	1	7 days
TKN-Auto Block Digest, Spect.	351.2	100 mL	P,G	1,3	28 days
Total Inorganic Carbon	415.1	(2) 40 mL	VOA vial	1	28 days
Total Inorganic Carbon if with TOC	415.1	(2) 40 mL	VOA vial	1	28 days
Total Organic Carbon-Oxidation	415.1	(2) 40 mL	VOA vial	1,3	28 days
Total Organic Halogen	9020	500 mL	Amber Glass	1,3	28 days
Turbidity	180.1	100 mL	P,G	1	48 hours
ELEMENTAL ANALYSES					
Chromium, Hexavalent	7196/6010	500 mL	P,G	1,9	24 hrs
GFAA(Furnace) Elements	SM 3113/ 200 series	500 mL	P,G	4	6 months
ICP Elements	200.7/6010	500 mL	P,G	4	6 months
ICP MS Elements	200.8/6020	500 mL	P,G	4	6 months
Low Level Mercury	1631	500 mL	G	NA	90 days
Mercury	245.1/7470	500 mL	P,G	4	28 days
GC ORGANIC ANALYSES					
BTEX & MTBE	602 & 8021	(2) 40 mL	VOA vial	1,8,9	14 days(~)
EDB, DBCP & 1,2,3-TCP	504.1	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Extractable Petroleum Hydrocarbons	MADEP/EPH	(2) 1000 mL	Amber Glass	12	14days/40days
Formaldehyde	556	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Fuel Oil in Water	8015Modified	(2) 1000 mL	Amber Glass	1,8	7days/40days
Fuel Oil in Water	ME HETL 4.1.25	(2) 1000 mL	Amber Glass	1,8	7days/40days
Gasoline in Water	8015Modified	(2) 40 mL	VOA vial	1,8	14 days
Gasoline in Water	ME HETL 4.2.17	(2) 40 mL	VOA vial	1,8	14 days
Glycols	8015Modified	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Herbicides	8151	(2) 1000 mL	Amber Glass	1	7days/ 40days
Methane, Ethane & ethene	RSK 175	(2) 40 mL	VOA vial	1,8,9	14 days(~)
PCB's (& Congeners)	608 & 8082	(2) 1000 mL	Amber Glass	1	7days/40days

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

TABLE 1 (cont.)

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER – AQUEOUS MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
GC ORGANIC ANALYSES					
Pesticides	608 & 8081	(2) 1000 mL	Amber Glass	1	7days/40days
Pesticides and PCB's	608 & 8081/8082	(2) 1000 mL	Amber Glass	1	7days/40days
Purgeable Aromatics	602 & 8021	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Purgeable Halocarbons	601 & 8021	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Purgeables, Total	601 & 602	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Purgeables, Total	8021	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Solvents (Direct Injection)	8015M	(2) 40 mL	VOA vial	1	14 days
Volatile Petroleum Hydrocarbons	MADEP/VPH	(2) 40 mL	VOA vial	11	14days
GC/MS ORGANIC ANALYSES					
Acid Extractables-Priority Pollutants	625	(2) 1000 mL	Amber Glass	1	7days/40days
Acid Extractables-TCL	8270	(2) 1000 mL	Amber Glass	1	7days/40days
Base Neutral Extract.-Priority Pollutants	625	(2) 1000 mL	Amber Glass	1	7days/40days
Base Neutral Extractables-TCL	8270	(2) 1000 mL	Amber Glass	1	7days/40days
Drinking Water Volatiles - Low Level	524.2	(3) 40 mL	VOA vial	1,8,9,10	14 days(~)
PCB Homologues	680	(2) 1000 mL	Amber Glass	1	7days/40days
Polyaromatic Hydrocarbons	8270/8270 SIM	(2) 1000 mL	Amber Glass	1	7days/40days
Semivolatile Extractables-Priority Pollutants	625	(2) 1000 mL	Amber Glass	1	7days/40days
Semivolatile Extractables-TCL	8270	(2) 1000 mL	Amber Glass	1	7days/40days
Volatile Organics	8260	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Volatile Organics-Priority Pollutants	624	(2) 40 mL	VOA vial	1,8,9	14 days(~)
HPLC ANALYSES					
HPLC-Explosives	8330, 8332	(2) 1000 mL	Amber Glass	1	7days/40days
MICROBIOLOGICAL ANALYSES					
Coliform, Fecal	SM 9222D, SM 9213D Mod.	100 mL	P,G	1,6	6 hours
Coliform, Total	SM 9222B	100 mL	P,G	1,6	30 hours
Coliform and E-coli, Total	SM9223B/Colitag	100 mL	P,G	1,6	30 hours
E-coli	SM9213D, Colilert/Quantitray	100 mL	P,G	1,6	6 hours
Heterotrophic Plate Count	SM9215B SIMPLATE	100 mL	P,G	1,6	30 hours

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

TABLE 1 (cont.)

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER – SOLID MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
GENERAL CHEMICAL ANALYSES		**4 oz=100 g**			
% Carbon	9060 mod.	4 oz	Soil Jar	1	28 days
Ammonia-Nitrogen-Automated Phenate	350.1 mod.	4 oz	Soil Jar	1	28 days (^)
Anions	9056	4 oz	Soil Jar	1	48hrs to 28 days from slurry (^)
Cation Exchange Capacity	9081	4 oz	Soil Jar	1	14days/7days (^)
Chloride-Automated Ferricyanide	9251/300.0	4 oz	Soil Jar	1	28days from slurry (^)
Cyanide, Amenable-Spectrophotometric	9012	4 oz	Soil Jar	1	14 days
Cyanide, Total-Spectrophotometric	9012	4 oz	Soil Jar	1	14 days
Fluoride, Potentiometric ISE	300.0 mod./340.2	4 oz	Soil Jar	1	28 days (^)
Lime Equivalency	310.1 mod.	4 oz	Soil Jar	1	28 days (^)
Nitrate+Nitrite-Automated Cadmium Reduction	300.0 mod./353.2	4 oz	Soil Jar	1	28 days (^)
Nitrate-Automated Cadmium Red./Diazotization	300.0 mod./353.2	4 oz	Soil Jar	1	48 hrs from slurry (^)
Nitrite-Automated Diazotization	300.0 mod./353.2	4 oz	Soil Jar	1	48 hrs from slurry (^)
Oil & Grease-Total Recoverable, Gravimetric N-Hexane extractable material N-Hexane extractable material w/ silica gel cleanup	9071	4 oz	Soil Jar	1	28 days (^)
Organic Nitrogen-Auto. Block Digest.,Spectro.	350.1/351.2 mod.	4 oz	Soil Jar	1	28 days (^)
pH (Laboratory)	9045	4 oz	Soil Jar	1	24 hours (^)
Phenolics, Total Recoverable-Manual 4AAP	Mod. 9065	4 oz	Soil Jar	1	28 days (^)
Phosphate, Ortho- Ascorbic Acid	300.0 mod./365.2	4 oz	Soil Jar	1	48 hrs from slurry (^)
Phosphate,Tot.-Auto Ascorbic Acid/Block Dig.	Mod. 365.4	4 oz	Soil Jar	1	28 days (^)
Solids-Ash	SM 2540 F	4 oz	Soil Jar	1	28 days (^)
Solids-Total Solids	CLP-CIP	4 oz	Soil Jar	1	28 days (^)
Solids-Volatile Solids	SM 2540 F	4 oz	Soil Jar	1	28 days (^)
Specific Conductance-Wheatstone Bridge	Mod. 9050	4 oz	Soil Jar	1	28 days (^)
Sulfate-Turbidimetric	9036/9038	4 oz	Soil Jar	1	28 days from slurry (^)
Sulfide-Iodometric	9030	4 oz	Soil Jar	1	7days from slurry (^)
Sulfide-Monier-Williams	40CFR-425	4 oz	Soil Jar	1	28 days (^)
Sulfite-Titrimetric	ASTM D3987/377.1 mod.	4 oz	Soil Jar	1	24 hrs from slurry (^)
TKN-Auto Block Digest,Spectro.	351.2 mod.	4 oz	Soil Jar	1	28 days (^)
Total Organic Halogen	9020/9021	4 oz	Soil Jar	1	28 days (^)
Total Petroleum Hydrocarbons-Extraction, IR	9071	4 oz	Soil Jar	1	28 days (^)
ELEMENTAL ANALYSES					
ICP Elements	6010	4 oz	Soil Jar	1	6 months
ICP MS ELements	6020	4 oz	Soil Jar	1	6 months
GFAA(Furnace) Elements	7000series	4 oz	Soil Jar	1	6 months
Mercury	7471	4 oz	Soil Jar	1	28 days

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

TABLE 1 (cont.)

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER – SOLID MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
ELEMENTAL ANALYSES **(cont.)**		**4 oz=100 g**			
Chromium, Hexavalent	3060/7196	4 oz	Soil Jar	1	30dys/24hrs
GC ORGANIC ANALYSES					
BTEX & MTBE	8021	(2) 40 mL	VOA Vial	1	14 days
Explosives - HPLC	8330, 8332	4 oz	Soil Jar	1	14days/40days
Extractable Petroleum Hydrocarbons	MADEP/EPH	4 oz	Soil Jar	1	7days/40days
Fuel Oil	ME HETL 4.1.25	4 oz	Soil Jar	1	14days/40days
Fule Oil	8015 mod.	4 oz	Soil Jar	1	14days/40days
Gasoline	ME HETL 4.2.17	(2) 40 mL	VOA Vial	1	14 days
Gasoline	8015 mod.	(2) 40 mL	VOA Vial	1	14 days
Herbicides	8151	4 oz	Soil Jar	1	14days/40days
PCB's (& Congeners)	8082	4 oz	Soil Jar	1	14days/40days
PCB's in Oil	8082	4 oz	VOA Vial	1	40 days
Pesticides	8081	4 oz	Soil Jar	1	14days/40days
Pesticides and PCB's	8081/8082	4 oz	Soil Jar	1	14days/40days
Purgeable Aromatics	8021	(2) 40 mL	VOA Vial	1	14 days
Purgeable Halocarbons	8021	(2) 40 mL	VOA Vial	1	14 days
Purgeables, Total	8021	(2) 40 mL	VOA Vial	1	14 days
Solvents (Direct Injection)	8015M	(2) 40 mL	VOA Vial	1	14 days
Volatile Petroleum Hydrocarbons	MADEP/VPH	(2)40 mL	VOA vial	13	28days
HPLC ANALYSES					
HPLC-Explosives	8330, 8332	4 oz	Soil Jar	1	7days/40days
GC/MS ANALYSES					
Acid Extractables-Priority Pollutants	8270	4 oz	Soil Jar	1	14 days/40 days
Acid Extractables-TCL	8270	4 oz	Soil Jar	1	14 days/40 days
Base Neutral Extractables-Priority Pollutants	8270	4 oz	Soil Jar	1	14 days/40 days
Base Neutral Extractables-TCL	8270	4 oz	Soil Jar	1	14 days/40 days
Polyaromatic Hydrocarbons	8270/8270SIM	4 oz	Soil Jar	1	14 days/40 days
Semivolatile Extractables-Priority Pollutants	8270	4 oz	Soil Jar	1	14 days/40 days
Semivolatile Extractables-TCL	8270	4 oz	Soil Jar	1	14 days/40 days
Volatile Organics – High Soil (>200 ug/kg)	5035/8260	Please refer to Table 6-2	Encore or similar sampler or VOA Vial or soil jar	14	Extruded w/in 48 hrs. Analyzed w/in 14 days
Volatile Organics – Low Soil (<200 ug/kg)	5035/8260	Please refer to Table 6-2	Encore or similar sampler or VOA Vial	14 or 15	Extruded w/in 48 hrs. Analyzed w/in 14 days
Volatile Organics-Priority Pollutants	8260	(2) 40 mL	VOA Vial	1	14 days
Volatile Organics-TCL	8260	(2) 40 mL	VOA Vial	1	14 days

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

TABLE 1 (cont.)

SAMPLING AND PRESERVATION REQUIREMENTS

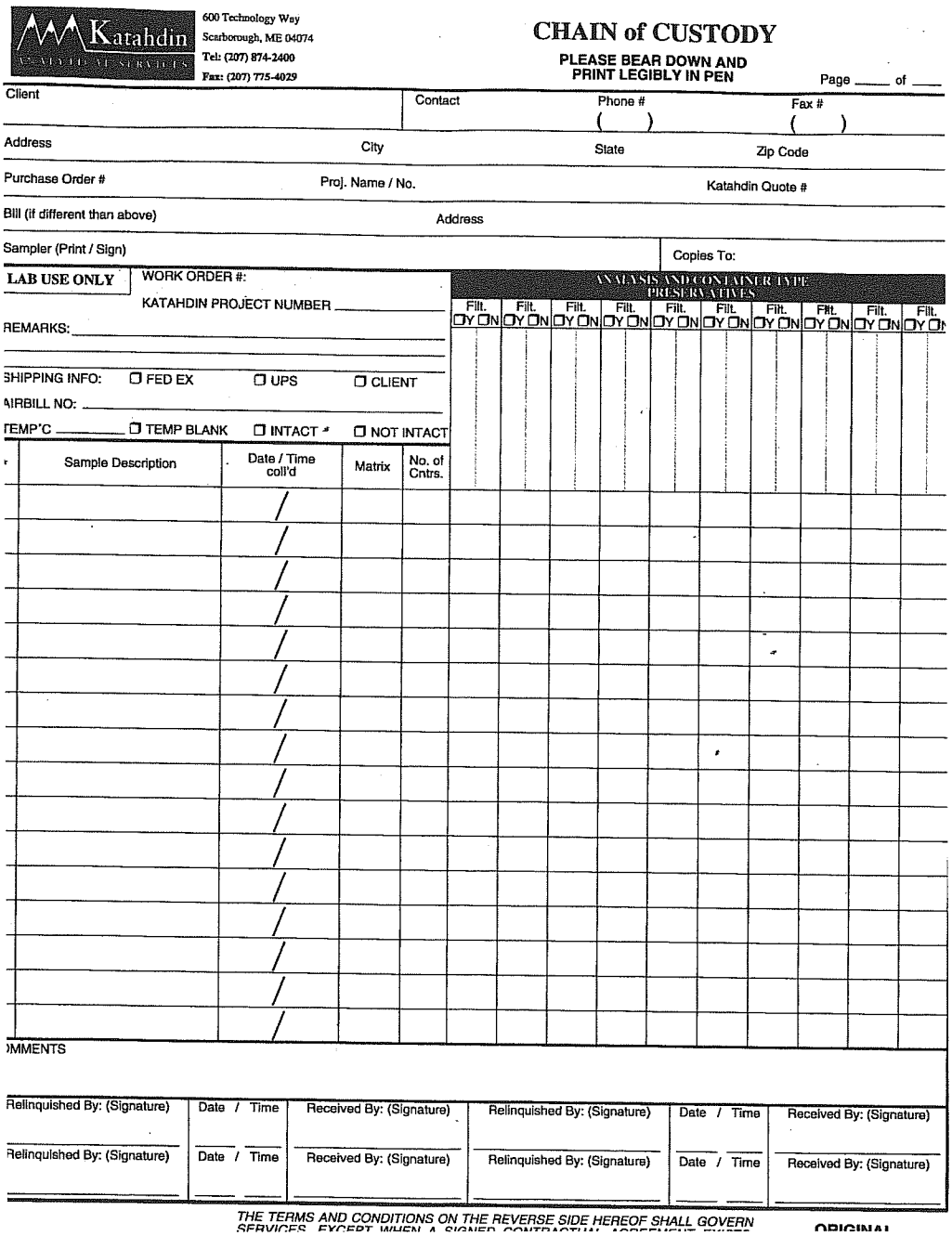
PARAMETER – SOLID MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
RCRA - HAZARDOUS WASTE CHARAC.					
Corrosivity-pH	9045	4 oz	Soil Jar	1	24 hours (^)
Ignitability-Flash Point (closed cup)	1010	4 oz	Soil Jar	1	14 days (^)
Reactivity-Reactive Cyanide	7.3.3.2	4 oz	Soil Jar	1	14 days
Reactivity-Reactive Sulfide	7.3.4.1	4 oz	Soil Jar	1	7 days
TCLP					
TCLP Extraction-Volatile Organics	1311	100 g	Soil Jar	1	14 days
TCLP Extraction-Semivolatiles	1311	200 g	Soil Jar	1	14 days
TCLP Extraction-Pesticides & Herbicides	1311	400 g	Soil Jar	1	14 days
TCLP Extraction-Metals	1311	200 g	Soil Jar	1	28 days
TCLP Analysis-Volatile Organics	8260	see above	Soil Jar	1	14 days
TCLP Analysis-Metals	6010/6020	see above	Soil Jar	1	180 days
TCLP Analysis-Mercury	7470	see above	Soil Jar	1	28 days
TCLP Analysis-Semivolatiles	8270	see above	Soil Jar	1	7 days/40 days
TCLP Analysis-Pesticides	8081	see above	Soil Jar	1	7 days/40 days
TCLP Analysis-Herbicides	8151	see above	Soil Jar	1	7 days/40 days

METHODS OF PRESERVATION
1 = Cool at 4 Degrees Celsius
2 = Settled
3 = H2SO4 to pH<2
4 = HNO3 to pH<2
5 = NaOH to pH>12
6 = 1 mL 0.1M Na2S2O3 or 1 10 mg pellet
7 = 1 m/L 2NZnAc/L & NaOH
8 = 2 drops 1:1 HCl
9 = No headspace
10 = Na2S2O3, if chlorinated
11 = HCl to pH < 2
12 = 5 mL of HCL
13 = 15 mL of methanol
14 = methanol
15 = sodium bisulfate

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 1

EXAMPLE OF STANDARD KATAHDIN CHAIN-OF-CUSTODY FORM



Katahdin
ANALYTICAL SERVICES
600 Technology Way
Scarborough, ME 04074
Tel: (207) 874-2400
Fax: (207) 775-4029

CHAIN of CUSTODY

PLEASE BEAR DOWN AND PRINT LEGIBLY IN PEN

Page ____ of ____

Client | Contact | Phone # ( ) | Fax # ( )

Address | City | State | Zip Code

Purchase Order # | Proj. Name / No. | Katahdin Quote #

Bill (if different than above) | Address

Sampler (Print / Sign) | Copies To:

LAB USE ONLY | WORK ORDER #:

KATAHDIN PROJECT NUMBER ____

REMARKS: ____

SHIPPING INFO: ☐ FED EX ☐ UPS ☐ CLIENT

AIRBILL NO: ____

TEMP'C ____ ☐ TEMP BLANK ☐ INTACT ☐ NOT INTACT

ANALYSIS AND CONTAINER TYPE / PRESERVATIVES

Sample Description	Date / Time coll'd	Matrix	No. of Cntrs.	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N
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COMMENTS

Relinquished By: (Signature)	Date / Time	Received By: (Signature)	Relinquished By: (Signature)	Date / Time	Received By: (Signature)
Relinquished By: (Signature)	Date / Time	Received By: (Signature)	Relinquished By: (Signature)	Date / Time	Received By: (Signature)

THE TERMS AND CONDITIONS ON THE REVERSE SIDE HEREOF SHALL GOVERN SERVICES. EXCEPT WHEN A SIGNED CONTRACTUAL AGREEMENT EXISTS.

ORIGINAL

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 2

EXAMPLE OF HOMEOWNER KATAHDIN CHAIN-OF-CUSTODY FORM

Katahdin
ANALYTICAL SERVICES

600 Technology Way
P.O. Box 540
Scarborough, ME 04070
Tel: (207) 874-2400 Fax: (207) 775-4029

Homeowner Chain of Custody

Client:	Contact:	Phone:	Fax:
Address:	City:	State:	Zip:
Purchase Order #:	Project Name/No.:	E-mail:	
Billing Address (if different):			
Sampler (Print/Sign):		Copies To:	
***Test results are for compliance and will be reported to the state (see statement below). yes no		Compliance samples must be received on ice.	
Lab Use Only	Work Order #:	KAS Project Manager:	Requested Services
Shipping: UPS Fed-Ex Mail Drop-Off			
Sample(s) Received on Ice? Yes No	Temperature if Iced:		

Sample Description (Sample Identification and/or Lot #)	Date Collected	Time Collected	No. of Cntrs.	Standard Homeowner	Arsenic	Total Coliforms	Lead	Safely Test – coliform & N+N	FHA/MSH	Fluoride	Uranium		What's Included in the Standard Test and the FHA/MSH Test.
													Standard Homeowner Total Coliform/e-coli Nitrate, Nitrite Chloride, pH Hardness Copper, Iron Manganese Sodium
													FHA/MSH Standard plus Lead Turbidity Color Odor

Relinquished By:	Date/Time:	Received By:	Relinquished By:	Date/Time:	Received By:

Per the National Environmental Laboratory Accreditation Program (NELAP) Standards, Katahdin is required to accept samples that have been properly preserved. All sample containers provided to you have been properly preserved, but the proper preservation also requires samples to be received at <6 degrees celcius. The Safe Drinking Water Act regulations only require this for compliance samples (i.e., results that are submitted to the state). By circling no for compliance (above), you acknowledge that the samples described above are not for compliance purposes, and thus may not meet the temperature receipt requirements.

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 3

EXAMPLE OF FOOD/MICROBIOLOGY KATAHDIN CHAIN-OF-CUSTODY FORM

Katahdin
ANALYTICAL SERVICES

600 Technology Way
P.O. Box 540
Scarbrough, ME 04070
Tel: (207) 874-2400 Fax: (207) 775-4029

Chain of Custody

Client:	Contact:	Phone:	Fax:
Address:	City:	State:	Zip:
Purchase Order #:	Project Name/No.:	E-mail:	
Billing Address (if different):			
Sampler (Print/Sign):		Copies To:	

Lab Use Only	Work Order #:	KAS Project Manager:	Food & Microbiological Services
Shipping: UPS Fed-Ex	Airbill No.:		
Temperature:			

Sample Description (Sample Identification and/or Lot #)	Date/Time Collected	Matrix	No. of Cntrs.	Plate Count (A/H/S)	Listeria	Yeast and Mold	Salmonella	E-Coli	E-Coli 0157:H7	Staph	Vibrio	Total Coliforms	Campylobacter	Shelf Life	Challenge Study		

Relinquished By:	Date/Time:	Received By:	Relinquished By:	Date/Time:	Received By:

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 4

EXAMPLE OF KATAHDIN SAMPLE RECEIPT LOGBBOK

KATAHDIN ANALYTICAL SERVICES, INC.

SAMPLE LOG IN

Date Received	Time Received	Date Logged In	Time Logged In	Work Order	Client	Initials
				SA 0094		
				SA 0095		
				SA 0096		
				SA 0097		
				SA 0098		
				SA 0099		
				SA 0100		
				SA 0101		
				SA 0102		
				SA 0103		
				SA 0104		
				SA 0105		
				SA 0106		
				SA 0107		
				SA 0108		
				SA 0109		
				SA 0110		
				SA 0111		
				SA 0112		
				SA 0113		
				SA 0114		
				SA 0115		
				SA 0116		
				SA 0117		
				SA 0118		
				SA 0119		
				SA 0120		
				SA 0121		
				SA 0122		
				SA 0123		
				SA 0124		

Signed By:_____ Date :_____

Reviewed By:_____ Date:_____

0000004

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 5

EXAMPLE OF WET CHEMISTRY SHORTS AND RUSHES LOGBOOK

WET CHEMISTRY SHORTS & RUSHES **Receipt Date:**

HOLDING TIME					Immediate			24 Hr		48 Hr								
Work Order / Client	Matrix	Earliest Sampling Date	Earliest Sampling Time	Rush Parameters	pH	DO	Sulfite	Fe +2	Cr+6	Total BOD	Carbon. BOD	Color	Nitrate	Nitrite	OPO4	Set. Solids	Turbidity	Comments (Quick TAT, MS/MSD, etc.)

0000004

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 6

EXAMPLE OF SAMPLE RECEIPT CONDITION REPORT FORM

Katahdin Analytical Services, Inc. **Sample Receipt Condition Report**

Client:	KAS PM:	Sampled By:
Project:	KIMS Entry By:	Delivered By:
KAS Work Order#:	KIMS Review By:	Received By:
SDG #:	Cooler: _____ of _____	Date/Time Rec.:

Receipt Criteria	Y	N	EX*	NA	Comments and/or Resolution
1. Custody seals present / intact?					
2. Chain of Custody present in cooler?					
3. Chain of Custody signed by client?					
4. Chain of Custody matches samples?					
5. Temperature Blanks present? If not, take temperature of any sample w/ IR gun.					Temp (°C):
Samples received at <6 °C w/o freezing?					Note: Not required for metals analysis.
Ice packs or ice present?					The lack of ice or ice packs (i.e. no attempt to begin cooling process) may not meet certain regulatory requirements and may invalidate certain data.
If not, has the cooling process begun (i.e. ice or packs present) and sample collection times <2hrs., but samples are not yet cool?					Note: No cooling process required for metals analysis.
6. Volatiles free of headspace: **Aqueous:** No bubble larger than a pea **Soil/Sediment:** Received in airtight container?					
Received in methanol?					
Methanol covering soil?					
7. Trip Blank present in cooler?					
8. Proper sample containers and volume?					
9. Samples within hold time upon receipt?					
10. Aqueous samples properly preserved? Metals, COD, NH3, TKN, O/G, phenol, TPO4, N+N, TOC, DRO, TPH – pH <2 Sulfide - >9 Cyanide – pH >12					

* Log-In Notes to Exceptions: document any problems with samples or discrepancies or pH adjustments

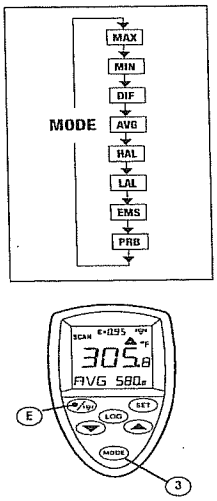
TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 7

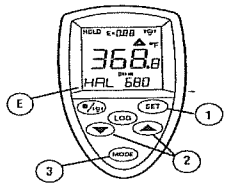
IR THERMOMETER MANUFACTURER'S INSTRUCTIONS FOR CHANGING EMISSIVITY

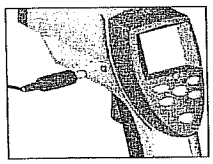
MODE Button Functions
Your infrared thermometer measures Maximum (MAX), Minimum (MIN), Differential (DIF)*, and Average (AVG)** temperatures each time you take a reading. This data is stored and can be recalled with the MODE button (3) until a new measurement is taken. (See "Hold and Recall" for information on how to recall stored data.) When the trigger is pulled again, the unit will begin measuring in the last mode selected. Pressing the MODE button also allows you to access the High Alarm (HAL), Low Alarm (LAL), Emissivity (EMS), Probe temperature (PRB—only available when the probe is connected), and Data logger (LOG). Each time you press MODE, you advance through the mode cycle. The diagram shows the sequence of functions in the Mode cycle.
Note: PRB (probe) is only available in the MODE loop when the contact probe is connected to the unit.

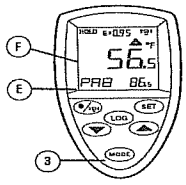
*DIF shows the difference between the maximum and minimum temperatures measured.
**AVG shows the average temperature reading for each time the trigger is pulled or the unit is locked on.



Selecting a Function
To Select the MAX, MIN, DIF, or AVG mode, pull the trigger. While holding the trigger, press the MODE button (3) until the appropriate code appears in the lower left corner of the display (E). Each time you press MODE, you advance through the MODE cycle. The MODE cycle is shown above.







Setting the High Alarm, Low Alarm, and Emissivity
To set values for the High Alarm (HAL), Low Alarm (LAL), and Emissivity, pull the trigger or press the MODE button (3) to activate the display. Press the MODE button until the appropriate code appears in the lower left corner of the display (E). Use the up and down keys (2) to adjust the desired values. To activate the alarms, press SET (1). To deactivate the alarms, press SET again.

Using a Probe (PRB)
Connect the probe to the input on the side of the unit (as shown). PRB automatically appears in the lower left corner of the display (E, below). The probe temperature is shown in the lower right part of the display. The current infrared temperature continues to show in the center of the display (F). While the probe is connected, you may still cycle through the mode functions by pressing MODE (3).

Note: PRB is only available in the MODE loop when a probe is connected to the unit; the probe temperature will not activate the high alarm or low alarm.

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 8

EXAMPLE OF KATAHDIN SAMPLE FILTRATION LOGBOOK

KATAHDIN ANALYTICAL SERVICES, INC.

Sample Filtration Logbook

Katahdin Sample No. List Individually	Site ID (Optional)	Filtration Requested By:		Filtered and Preserved By:		
		Initials	Date	Initials	Date	Time

Reviewed and Approved by: ____________________ **Date:** __________

QAAA097 **0000001**

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 9

MEASURED TURBIDITY AND PRESERVATION OF INCOMING SAMPLES LOGBOOK

KATAHDIN ANALYTICAL SERVICES

Measured Turbidity and Preservation of Incoming Samples

KAS Lab Sample ID	Measured Turbidity (NTU)	Turbidity Date	Turbidity Analyst	Preservation Date	Preservation Time	Preservation Analyst
WW6757-9A	0.22	12-19-06		12/14/06	~~1430~~ 1440	DM
↓ 10A	0.18	↓		↓	↓ ↓	↓
WW6758-1A	4.80	↓		↓	↓ ↓	↓
WW6776-1A	0.64	12-20-06		12/20/06	1000	DM
WW6855-1A	0.92	12-26-06		12/26/06	1600	DM
WW6860-1A	2.40	↓		↓	↓	↓
SA0027-1A	0.52	1-3-07		01/03/07	1445	DM
SA0028-1A	0.72	1-3-07		↓	↓	↓
SA0087-1A	0.26	1-5-07		01/05/07	1645	DM
SA0088-1A	10.4	1-5-07		↓	↓	↓
SA0110-1A	0.47	1-8-07		01/08/07	1430	DM
SA0109-1A	0.32	1-8-07		↓	↓	↓
SA0138-1A	0.12	1-04-07		01/04/07	1700	DM
SA0262-1A	0.14	1-18-07		01/18/07	0930	DM
SA0263-1A	0.33	1-18-07		↓	↓	↓
SA0267-1A	0.30	1-18-07		01/18/07	0950	DM
SA0313-1A	0.58	1-22-07		01/22/07	1030	↓
SA0316-1A	1.70	1-22-07		01/22/07	1345	DM
REVIEWED BY:			**DATE:**			

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 10

EXAMPLE OF LABORATORY INCOMING SAMPLE INFORMATION SHEET (ISIS)

KATAHDIN ANALYTICAL SERVICES, INC. – INCOMING SAMPLE INFORMATION SHEET

CLIENT:	ATTENTION:
ADDRESS:	
TELEPHONE:	FAX:
OTHER CLIENT CONTACTS:	
REPORT CC:	PM/SALES CONTACT:

PO #:	CLIENT ACCOUNT:
KAS QUOTE #:	KIMS QUOTE #:
PROJECT NAME:	KIMS PROJECT REF.:
SDG:	APPLICABLE CERTIFICATION:
RECEIPT DATE:	MEANS OF DELIVERY:

METHOD OR KIMS PRODUCT	MATRIX	# OF SAMPLES	SPECIAL INSTRUCTIONS (I.E LIMITS, PREPS)

SUBCONTRACT PARAMETER	SUBCONTRACT LABORATORY

QC LEVEL:	EDD:
RUSH/VERBAL TAT:	HARDCOPY TAT:
HISTORY:	
OTHER:	

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 11

EXAMPLE OF KATAHDIN WORK ORDER/LOGIN COC REPORT

Katahdin
ANALYTICAL SERVICES

Katahdin Analytical Services

Login Chain of Custody Report (lno1)

Jan. 26, 2007
03:51 PM

Page: 1 of 1

Login Number: SA0395

Account:KATAHD001 Web
Katahdin Analytical Services

Project:

Primary Report Address:
Leslie Dimond
Katahdin Analytical Services
600 Technology Way
P.O. Box 540
Scarborough,ME 04070

Primary Invoice Address:
Accounts Payable
Katahdin Analytical Services
600 Technology Way
P.O. Box 540
Scarborough,ME 04070

Report CC Addresses:
Invoice CC Addresses:

Login Information

ANALYSIS INSTRUCTIONS	:
CHECK NO.	:
CLIENT PO#	:
COOLER TEMPERATURE	: n/a
DELIVERY SERVICES	: In House
EDD FORMAT	:
MAIL DATE	:
PM	: LAD
PROJECT NAME	: QC Holding Blanks
QC LEVEL	: I
REGULATORY LIST	:
REPORT INSTRUCTIONS	:
SDG ID	:
SDG STATUS	:

Laboratory Sample ID	Client Sample Number	Collect Date/Time	Receive Date	PR	Verbal Date	Due Date	Comments
SA0395-1	WHITE FRIDGE	26-JAN-07 15:50	26-JAN-07			08-FEB-07	
Matrix	*Product*	*Hold Date (shortest)*	*Bottle Type*		*Bottle Count*		
Aqueous	S SW8260FULL5ML	09-FEB-07			2		
SA0395-2	BLUE FRIDGE	26-JAN-07 15:50	26-JAN-07			08-FEB-07	
Matrix	*Product*	*Hold Date (shortest)*	*Bottle Type*		*Bottle Count*		
Aqueous	S SW8260FULL5ML	09-FEB-07			2		

Total Samples: 2 ***Total Analyses:*** 2

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 12

EXAMPLE OF LABORATORY INTERNAL CHAIN-OF-CUSTODY FORM

Katahdin Analytical Services, Inc.
Internal Custody Record

EXTRACTIONS (AQ) Work Order #:_____

Sample Numbers Involved	Placed in refrigeration by: Initials
	Date
	Time

Sample Numbers	Analysis	Removed			Returned			Consumed?	
		Initials	Date	Time	Initials	Date	Time	Yes	No

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 13

EXAMPLE OF REFRIGERATOR TEMPERATURE LOGBOOK

KATAHDIN ANALYTICAL SERVICES, INC.

SAMPLE RECEIPT WALK-IN TEMPERATURE LOG

Corrective Action: Note in the "comments" column and notify the QAO or supervisor; document corrective actions taken and return to control.

Thermometer Location		Sample Receipt Walk-in 1	Comments
Acceptance Criteria		2 to 6 °C	
Date	Initials	Temp (°C)	
02/2/07	NDA	3.7	
02/05/07	DWM	5.8	
02/06/07	DWM	5.8	
02/07/07	DWM	5.8	
02/08/07	DWM	3.8	
02/09/07	DWM	3.5	
02/12/07	DWM	3.5	
02/13/07	DWM	3.5	

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 14

EXAMPLE OF IMMEDIATE INTERNAL COC LOGBOOK

KATAHDIN ANALYTICAL SERVICES, INC.
INTERNAL CUSTODY RECORD FOR IMMEDIATES

CLIENT	PROJECT	CLIENT ID &/or WORK ORDER #	ANALYSIS	OUT date/time	IN date/time	INIT	Consumed?
Jacobs		WW4813-1A, -2A	ICP	9/13/06 0930	→0935	DJJ	yes no
Jacobs		WW4883-1A	ICP	9/16/06 0100	→1000	DJJ	yes no
CES		WW4965	BOD	9/20/06 0830	9/20/06 1000	CP	yes no
CCAR		WW4969	BOD	9/20/06 1000	↓	CP	yes no
GEMF		WW4970	BOD	↓	↓	CP	yes no
Jacobs		WW4962-1A, -2A	ICP	9/20/06 0900	→1000	DJJ	yes no
Irving		WW4994	BOD	9/21/06 1000	9/21/06 1045	CP	yes no
Highlimer		WW4992 ②	BOD	9/21/06 1045		CP	yes no
NATIONAL		WW5000	TS, RESIDUE PH, SP.CONO	9/21/06 1100	9/22/06 1217	DJ	yes no
W+C		WW5001	BOD	9/21/06 1300		CP	yes no
Aliens		WW5016	NO7	9/22/06 1300	0 9/22/06 1100	KR	yes no
RAASOM		WW5010	↓	↓ ↓	↓	↓	yes no
EcoMaine		WW5029	BOD	9/20/06 1100		CP	yes no

QAQC143

0000095

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: SD-903
Revision History
Cover Page
Page 1

TITLE: **SAMPLE DISPOSAL**

Prepared By: _____ Date: 2/01

Approved By:

Group Supervisor: _____ Date: _____

Operations Manager: Jal C. Burton Date: 2/01

QA Officer: Deborah J. Nadeau Date: 2.01

General Manager: _____ Date: 2/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Format changes, ~~added pollution prevention~~, added updated log-book and greater detail on disposal.	DN	2.01	2/01
02	Major rewrite to include more detail on hazardous waste regulations & to reflect current practices.	DN	02/05	02/05
03	Rewrite of section 7 to comply with current practices in new facility. Updated Figures 1 to 3.	DN	02.08	02.08
04	Added elementary neutralization to section 7.0. Other minor edits.	DN	05.09	0509

TITLE: SAMPLE DISPOSAL

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SD-903-04**, titled **SAMPLE DISPOSAL**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SD-903-04**, titled **SAMPLE DISPOSAL**.

Recipient: _____ Date: _____

TITLE: SAMPLE DISPOSAL

1.0 SCOPE AND APPLICATION

Katahdin Analytical Services, Inc. requires strict adherence to specific procedures for the disposal of samples. The procedures are designed to categorize waste materials, provide for their safe and timely disposal and to ensure compliance with local and federal regulations pertaining to disposal of chemicals and environmental samples. Any other means of disposal not described in this SOP is prohibited without consent from the Katahdin Environmental Health & Safety Officer and/or the Katahdin Environmental Compliance Officer.

The purpose of this SOP is to describe the procedures utilized by Katahdin Analytical personnel for the disposal of samples. These procedures apply to the disposal of all samples received or processed by Katahdin. Refer to the current revision of Katahdin SOP CA-107 regarding the disposal of spent preparation and analysis reagents, standards, sample extracts, distillates, or digestates.

1.1 Definitions

Hazardous Waste – A "Solid Waste" which displays a hazardous characteristic or is specifically listed as hazardous waste.

Solid Waste – Any discarded material that is not excluded from the definition of hazardous waste.

Discarded Material – Material that is abandoned, recycled or inherently waste-like.

Waste (State of Maine) –

- Any useless, unwanted, or discarded substance or material, whether or not such substance or material has any other future use.
- Any substance or material that is spilled, leaked, pumped, poured, emptied or dumped onto the land or into the water or ambient air.
- Materials which are used in a matter constituting disposal, burned for energy recovery, reclaimed, or accumulated speculatively.

Ignitable Hazardous Waste – EPA Waste Code D001

- Liquids with a flash point less than 140°F or 60°C.
- Solids capable of spontaneous combustion under normal temperature and pressure.
- Ignitable compressed gas.
- Oxidizers.

Corrosive Hazardous Waste - Liquids with a pH less than or equal to 2.0 or greater than or equal to 12.5. EPA waste code D002.

Reactive Hazardous Waste – EPA waste code D003.

- A material that reacts violently with water.
- A material that generates toxic gases or fumes.
- Explosives.

Toxic Hazardous Waste – A material that exceeds certain concentration levels based on the toxicity characteristic leaching procedure (TCLP). See Figure 3 for the chemicals and concentration levels covered under this definition.

Listed Wastes – Lists of chemicals that are considered hazardous based on the following criteria

- Virgin chemical or unused product.
- Sole active ingredient.
- Single substance spill debris.

Listed wastes are divided into 5 subcategories

- F-wastes – Describe hazardous waste from non-specific sources usually containing halogenated and non-halogenated solvents.
- K-wastes – Describe hazardous wastes created by specific processes.
- U-wastes – Describe toxic or non-acute hazardous wastes.
- P-wastes – Describe acute hazardous wastes. (Note: Maine considers a material to be a P-listed waste if it contains 10% or more of any P-listed chemical.
- State listed wastes – Maine lists any material with a concentration of greater than 50 ppm Polychlorinated Biphenyls (PCB) as a hazardous waste.

Organics hit – A liquid sample containing greater than 1 mg/L of organic contaminants or a soil sample containing greater than 20 mg/kg of organic contaminants.

1.2 Responsibilities

Only designated analysts/technicians trained in these procedures may dispose of samples or analytical by-products. Each analyst or technician must be familiar with Katahdin Analytical safety procedures. Gloves, safety glasses, lab coats and/or other protective clothing must be worn at all times.

It is the responsibility of the designated Katahdin personnel involved in the disposal of samples to read and understand this SOP, to adhere to the procedures outlined, to properly document their activities in the appropriate lab notebook and file the necessary manifests and reports to outside agencies in the required manner. Refer to

Katahdin SOP QA-805, "Personnel Training & Documentation of Capability," current revision.

It is the responsibility of the Department Managers to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

It is the responsibility of the Katahdin Environmental Health & Safety Officer (EHSO) to manage the proper classification and disposal of samples. Katahdin is responsible for regulatory compliance of Katahdin's waste storage areas (less than 90 day storage). The EHSO ensures compliance of the waste storage areas with applicable state and federal regulations. The EHSO is responsible for providing the appropriate training to all individuals involved in the proper classification and/or disposal of samples. The EHSO is responsible for working with the Laboratory Operations Manager/Environmental Compliance Officer to help identify problems and assure resolution, to facilitate corrective action where needed, and to communicate unresolved problems and concerns to the Laboratory Vice President.

It is the responsibility of the Operations Manager/Environmental Compliance Officer to oversee adherence to Katahdin sample disposal and hazardous waste practices by all laboratory groups under his/her authority, to help identify problems and assure resolution, to facilitate corrective action where needed, and to communicate problems and concerns to the EHSO and/or the Laboratory Vice President.

It is the responsibility of the Laboratory Vice President to provide the necessary resources to meet the regulatory requirements of proper classification and disposal of samples.

2.0 SUMMARY OF METHOD

Not applicable.

3.0 INTERFERENCES

Not applicable.

4.0 APPARATUS AND MATERIALS

Not applicable.

TITLE: SAMPLE DISPOSAL

5.0 REAGENTS

Not applicable.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Not applicable.

7.0 PROCEDURES

7.1 Sample purging is the removal of samples from laboratory refrigerated storage. Sample storage areas where samples are removed (purged) from include wet chemistry, organic extractables, metals, volatiles, total organic carbon and soils. Wet chemistry, aqueous metals, organic extractables, total organic carbon, and soils can all be found in the walk-in refrigerator. Aqueous and soil volatiles can be found in the volatiles laboratory refrigerators/freezer.

7.2 Samples are purged from storage, after analysis and reporting, on a routine basis to make room for incoming samples. Samples are to be kept in storage for a duration of 30 days past the report mailed date. Some samples must be kept for 60 or 90 days beyond the report mailed date, depending on specific client requests and contracts.

7.3 The first step in disposing of samples is to generate a disposal list. The disposal list contains sample analysis information stored in the Katahdin Information Management System (KIMS). The analytical data for the samples is compared to the hazardous waste criteria specified in 40CFR Part 261 and to local wastewater discharge criteria. Refer to Figure 4 for 40 CFR Part 261 Characteristic Hazardous Waste Criteria. Based on this comparison, the report displays information on the classification/category for disposal of each sample. The disposal report should be reviewed against the data reports for accuracy. Refer to Figure 2 for an example of a KIMS generated disposal list. The primary disposal categories listed in the report are: non-hazardous, high organics, high metals, flashpoint, high mercury, high PCBs, and high cyanide. Katahdin has established 14 waste stream profiles with a 3rd party waste transporter/waste disposal firm for sample disposal based on these categories. As required, new or special temporary waste profiles are established based on the characteristics of samples.

7.4 Sorting through samples and preparing them for disposal is a crucial quality checkpoint. Samples put into the incorrect waste stream could not only produce adverse environmental effects, but, could also interrupt the 3rd party's waste treatment efficiency, or endanger an individual handling the waste stream. Therefore, when sorting through samples pay close attention to which waste stream each sample falls into.

7.5 Once you are ready to dispose of the samples of interest (the oldest samples that have been purged), these samples must be sorted, logged, and the classification/category (sample knowledge) information recorded.

Sample storage times (as listed in section 7.2) and space should be taken into consideration when purging samples. It is important to make room for future samples, but to make sure that samples are not purged too early. Samples should be pulled from the walk-in or the volatiles refrigerators to make room for new samples. When purging, chose a section that needs extra space the most and remove the oldest samples.

Safety glasses, nitrile gloves, lab coat, and a splash apron must be worn when handing samples during disposal

7.6 Remove the designated purge samples from the shelf one by one and line them up on the countertop in the log-in area. Generally, removing two cartloads at a time is a good amount to purge at one time. For volatile samples in 40mL vials, 5 or 6 vial trays should be purged at a time. Samples should be lined up across the counter with the earliest sample to the left and building up to the right, organizing the samples according to work order and sample number. After the samples are lined up, they should be recorded in the Sample Disposal Logbook (SDL). Refer to Figure 1 for an example SDL page. The location the samples were removed from should also be recorded. Sample storage areas are recorded with the following designations:

VOA (Aq)	Aqueous Volatiles(VOA)
VOA (SL)	Solid Volatiles(VOA)
M	Metals
EXT	Extractables (Organic)
TOC	Total Organic Carbon
WC	Wet Chemistry
S	Soils

7.7 The next step is to use the sample disposal list to determine the earliest release date of the reports and to determine each samples appropriate waste classification/characterization. As stated in section 7.3, the primary disposal categories listed in the report are: non-hazardous, high organics, high metals, flashpoint, high mercury, high PCBs, and high cyanide.

Using the information from the KIMS disposal list, record the appropriate classification for each sample in the SDL. If multiple categories are identified as being present then a single category is selected as controlling. The order of precedence is PCB's, metals and then organics. If another scenario is found, the individual should bring it to the EHSO for a determination of the acceptable waste stream designation or a determination that it should be lab packed separately.

If samples have been sorted that have not been in storage for the 30 days beyond the release date (60 or 90 for certain clients), then these samples need to be placed back in storage and it should be noted in the SDL.

7.8 As stated above, a sample may be categorized into a waste stream based upon the analytes it contains as determined by laboratory testing. In addition, many samples are also categorized as hazardous waste based upon the preservative that they contain. Since many samples contain preservatives, caution must be used when dumping samples. It is also important to ensure that the sample container is empty. This can be accomplished by holding the container upside down and shaking gently until liquid is no longer observed coming out of the container.

7.9 Once waste categories have been determined and entered into the SDL, The following waste categories are disposed of as follows:

7.9.1 Dumping non-hazardous samples (as determined by laboratory testing)

Non-hazardous samples (non-preserved) are poured directly into the sink in the warehouse.

Non-hazardous solid samples are disposed of with the general trash, which is picked up by commercial trash collectors and ultimately disposed of in a waste-to-energy incinerator.

Sample containers from non-hazardous samples are disposed of with the general trash.

7.9.2 Dumping Samples with high Organics (as determined by laboratory testing)

Aqueous samples get dumped into waste stream "K". Containers are disposed of with general trash. Solid samples are placed into waste stream "I" with their containers. The disposal date is recorded in the SDL.

7.9.3 Dumping samples high in metals, including mercury (as determined by the by laboratory testing)

Aqueous samples get disposed of in waste stream "A". Containers are disposed of with general trash. Solid samples are placed in waste stream "L" with their containers. The disposal date is recorded in the SDL.

7.9.4 Dumping Acidic Samples that do not contain any other hazardous waste constituents (as determined by the acidic preservative or by laboratory testing)

Refer to section 7.10 below.

7.9.5 Dumping Basic samples (as determined by the basic preservative or by laboratory testing)

Aqueous samples get disposed of in waste stream "NHi". Containers are disposed of with general trash. The disposal date is recorded in the SDL.

7.9.6 Dumping samples with high PCBs (as determined by laboratory testing)

Aqueous samples are disposed of in waste stream "Q". Containers are disposed of with general trash. Solid samples get disposed of in waste stream "F" with their containers. The disposal date is recorded in the SDL.

7.9.7 Dumping samples with low flashpoints (as determined by laboratory testing)

Aqueous samples are disposed of in waste stream "O". Containers are disposed of with general trash. Solid samples get disposed of in waste stream "I" with their containers. The disposal date is recorded in the SDL.

7.9.8 Dumping samples with high cyanide (as determined by laboratory testing)

Aqueous samples are disposed of in waste stream "NHi". Containers are disposed of with general trash. Solid samples should be set aside for labpack. The disposal date is recorded in the SDL.

7.9.9 Miscellaneous Disposal (as determined by the preservative)

Sodium Bisulfate: Sodium Bisulfate often comes in vials, but may also come in the 2-4oz glass jars. Dump the Sodium Bisulfate out of the container into waste stream "A". There may be remaining soil left in the sample container. The soil's waste stream and dump date will be dictated by the SDL. The disposal date is recorded in the SDL.

Methanol / Free Products: This often comes in vials, but may also come in the 2-4oz glass jars. Dump the methanol out of the container into the mix-flammables accumulation. When this satellite accumulation container gets full it can be dumped into the "O" waste stream. There may be remaining soil left in the sample container. The soil's waste stream and dump date will be dictated by the SDL. Lastly, samples marked "free product" on the Katahdin sample ID label can be dumped into the mixed flammables stream. The disposal date is recorded in the SDL.

7.10 Pursuant to Maine DEP regulations, Katahdin has the necessary agreements, processes and documentation in place to neutralize samples without a license. Refer to the current revision of the Katahdin Environmental Health & Safety Manual for additional information. Generally, the following procedures are followed.

7.10.1 Samples that have been determined to be hazardous due **solely** to the corrosivity characteristic are neutralized using sodium hydroxide pellets. In the warehouse, samples are emptied into a five gallon heavy duty carboy to about 60% capacity. The carboy is kept in a secondary container. Sodium hydroxide pellets are added slowly to the carboy (about 5 grams at a time) and stirred with a long glass stirring rod. The pH is checked with pH paper.

7.10.2 This process is continued until the pH is between 7 and 8. This normally takes about 30-40 grams of sodium hydroxide pellets, but may vary depending on the buffering capacity of the individual samples.

7.10.3 The carboy is emptied into the sink in the warehouse. The tap water is run at the same time as the neutralized material is disposed of. An eyewash station and spill material is located at this sink.

7.10.4 All neutralization activities are documented, including the date and time of neutralization, the name of the person doing the neutralizing, the amount of neutralized liquid discharged, details on the inspection of the drain area and the date and nature of any significant repairs or corrective actions. This documentation is maintained by the EHSO. Refer to Figure 5 for an example logbook page of neutralization documentation.

7.11 Every 3 to 5 weeks a pickup of hazardous waste is scheduled with the 3rd party waste transporter/waste disposal firm. An inventory is faxed to the transporter summarizing the number of drums and waste streams/profiles. As required, a "lab pack" of expired chemicals or orphan samples is organized as necessary. A designated individual, with applicable Hazardous Waste (RCRA) and Department of Transportation (DOT) training, oversees the waste pickup and signs the hazardous manifests and land ban documentation. Within 7 days a copy is forwarded to the Maine Department of Environmental Protection (MEDEP) and the environmental agency in the designation state (if required by that state). Once the report is received at the disposal facility a copy is returned to KATAHDIN and the MEDEP.

7.12 Prior to March 31 of each year, the laboratory prepares the Annual Hazardous Waste Report (i.e., MEDEP modified EPA Form 8700-13A) as required by MEDEP Hazardous Waste Management Rules. The complete report is reviewed by the Katahdin Environmental Compliance Officer and then forwarded to the following address:

Maine Department of Environmental Protection
Bureau of Remediation & Waste Management
State House Station #17
Augusta, ME. 04333
Attn: Annual Hazardous Waste Report

TITLE: SAMPLE DISPOSAL

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

On a daily basis, a designated individual performs quality checks in all hazardous waste storage areas. The daily check documentation is located in login. Any discrepancy is copied to the Operations Manager and the Katahdin Vice President for corrective action. Refer to the current revision of Katahdin SOP CA-107, *The Management of Hazardous Waste as it Relates to the Disposal of Laboratory Process Waste, Reagents, Solvents & Standards*, for more information. Refer to Figure 3 for a copy of the daily check documentation.

9.0 METHOD PERFORMANCE

Not applicable.

10.0 APPLICABLE DOCUMENTS/REFERENCES

USEPA Code of Federal Regulations, 40 CFR Part 261.

Maine Department of Environmental Protection (ME DEP) Hazardous Waste Management Rules

ME DEP modified EPA Form 8700-13A

LIST OF TABLES AND FIGURES

TITLE: SAMPLE DISPOSAL

FIGURE 1

EXAMPLE OF SAMPLE DISPOSAL LOGBOOK (SDL)

KATAHDIN ANALYTICAL SERVICES, INC. -SAMPLE STORAGE/DISPOSAL LOGBOOK

WORK ORDER/ SAMPLE NUMBERS	DEPARTMENT	EARLIEST RELEASE DATE	< CRITERIA	SAMPLE KNOWLEDGE									DATE DISPOSED	INITIALS
			CLEAN	WL	ORG	METS	CN	FP	HG	PCBS				
SA5783-1	WC	10-17-07	✓										1-22-08	GN
SA5786-1		10-17-07	✓											
SA5787-1,2,4		10-17-07	✓											
SA5790-1		10-19-07		✓										
SA5793-1		10-17-07	✓											
SA5795-1-9		10-23-07	✓											
SA5797-1		10-23-07	✓											
SA5798-1,2		10-25-07			✓									
SA5799-1-5		10-31-07	✓											
SA5804-1,2		10-25-07	✓											
SA5809-1,2		10-25-07	1			2				2				
SA5810-1-4		10-25-07	✓											

QAQC354

0000022

TITLE: SAMPLE DISPOSAL

FIGURE 2

EXAMPLE OF KIMS GENERATED WASTE DISPOSAL REPORT

SAMPLE DISPOSAL REPORT

Query by: Login SA6501 to SA7000
Date : 15-JAN-08

Sample	SDG	Status	Mail Date	Parameter	Value		
SA6605-1		NEED	12/02/07				
SA6606-1		NEED	12/02/07				
SA6607-1		NEED	11/15/07				
SA6608-1		NEED	12/06/07				
				ORG	1.17	MG/L	(HIGH)
SA6608-1		NEED	12/06/07				
SA6608-2		NEED	12/06/07				
				AA	13	MG/KG	(HIGH)
SA6609-1		NEED	11/26/07				
SA6609-1		NEED	11/26/07				
SA6610-1		NEED	11/30/07				
SA6611-1	FCS-020	NEED	12/07/07				
SA6611-2	FCS-020	NEED	12/07/07				
SA6611-3	FCS-020	NEED	12/07/07				
SA6611-4	FCS-020	NEED	12/07/07				
SA6611-5	FCS-020	NEED	12/07/07				
SA6611-6	FCS-020	NEED	12/07/07				
SA6611-7	FCS-020	NEED	12/07/07				
SA6611-8	FCS-020	NEED	12/07/07				
SA6612-1	NSA-030	NEED	12/07/07				
SA6612-2	NSA-030	NEED	12/07/07				
SA6612-3	NSA-030	NEED	12/07/07				
SA6612-4	NSA-030	NEED	12/07/07				
				ORG	1.70735	MG/L	(HIGH)
SA6612-5	NSA-030	NEED	12/07/07				
				ORG	1.0481	MG/L	(HIGH)

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TITLE: SAMPLE DISPOSAL

FIGURE 3

EXAMPLE OF HAZARDOUS WASTE STORAGE AREA DAILY CHECK

Daily Checklist for
HAZARDOUS WASTE STORAGE AREA

Week of: 1-28, 20 03

Item/Day:	Monday	Tuesday	Wednesday	Thursday	Friday
1. Are containers closed? (Except when waste is being added)	(Yes) / No	(Yes) / No	(Yes) / No	(Yes) / No	(Yes) / No
2. Are containers properly labeled with a hazardous waste label?	(Yes) / No	(Yes) / No	(Yes) / No	(Yes) / No	(Yes) / No
3. Do you have access to each container and can you read the label? (36" aisle?)	(Yes) / No	(Yes) / No	(Yes) / No	(Yes) / No	(Yes) / No
4. Is each container marked with the date storage began?	(Yes) / No	(Yes) / No	(Yes) / No	(Yes) / No	(Yes) / No
5. Are the dates on the containers less than 90 days old?	(Yes) / No	(Yes) / No	(Yes) / No	(Yes) / No	(Yes) / No
6. Is container free of dents, bulges, rust, spills or leaks?	(Yes) / No	(Yes) / No	(Yes) / No	(Yes) / No	(Yes) / No
7. Are all containers on a firm working surface?	(Yes) / No	(Yes) / No	(Yes) / No	(Yes) / No	(Yes) / No
8. Inspection by: Name (No Initials)	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
9. Time of Inspection	16:35	15:00	14:45	14:15	16:25
10. Verification of Inspection (Name/Date)	[illegible] 1-28-03	[illegible] 1-29-03	[illegible] 1-30-03	[illegible] 1-31-03	[illegible] 1-27-03

Deficiency noted:					
Corrective action:					
By (Name/Date):					

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0900060

TITLE: SAMPLE DISPOSAL

FIGURE 4

CHARACTERISTIC TOXIC HAZARDOUS WASTE AND TCLP CONCENTRATIONS

Chemical Name	CAS Number	Waste Code	TCLP conc. liquid	Equivalent conc. In Soil
Arsenic	7440-38-2	D004	5.0 mg/L	100 mg/kg
Barium	7440-39-3	D005	100 mg/L	2000 mg/kg
Cadmium	7440-43-9	D006	1.0 mg/L	20 mg/kg
Chromium	7440-47-3	D007	5.0 mg/L	100 mg/kg
Lead	7439-92-1	D008	5.0 mg/L	100 mg/kg
Mercury	7439-97-6	D009	0.2 mg/L	4 mg/kg
Selenium	7782-49-2	D010	1.0 mg/L	100 mg/kg
Silver	7440-22-4	D011	5.0 mg/L	20 mg/kg
Endrin	72-20-8	D012	0.02 mg/L	0.4 mg/kg
Lindane	58-89-9	D013	0.4 mg/L	8 mg/kg
Methoxychlor	72-43-5	D014	10 mg/L	200 mg/kg
Toxaphene	8001-35-2	D015	0.5 mg/L	10 mg/kg
2,4-D	94-75-7	D016	10 mg/L	200 mg/kg
2,4,5-TP (Silvex)	93-72-1	D017	1.0 mg/L	20 mg/kg
Benzene	71-43-2	D018	0.5 mg/L	10 mg/kg
Carbon Tetrachloride	56-23-5	D019	0.5 mg/L	10 mg/kg
Chlordane	57-74-9	D020	0.03 mg/L	0.6 mg/kg
Chlorobenzene	108-90-7	D021	100 mg/L	2000 mg/kg
Chloroform	67-66-3	D022	6.0 mg/L	120 mg/kg
o-Cresol	95-48-7	D023	200 mg/L	4000 mg/kg
m-Cresol	108-39-4	D024	200 mg/L	4000 mg/kg
p-Cresol	106-44-5	D025	200 mg/L	4000 mg/kg
Cresol	1319-77-3	D026	200 mg/L	4000 mg/kg
1,4-Dichlorobenzene	106-46-7	D027	7.5 mg/L	150 mg/kg
1,2-Dichloroethane	107-06-2	D028	0.5 mg/L	10 mg/kg
1,1-Dichloroethylene	75-35-4	D029	0.7 mg/L	14 mg/kg
2,4-Dinitrotoluene	121-14-2	D030	0.13 mg/L	2.6 mg/kg
Heptachlor	76-44-8	D031	0.008 mg/L	0.16 mg/kg
Hexachlorobenzene	118-74-1	D032	0.13 mg/L	2.6 mg/kg
Hexachlorobutadiene	87-68-3	D033	0.5 mg/L	10 mg/kg
Hexachloroethane	67-72-1	D034	3.0 mg/L	60 mg/kg
Methyl Ethyl Ketone	78-93-3	D035	200 mg/L	4000 mg/kg
Nitrobenzene	98-95-3	D036	2.0 mg/L	40 mg/kg
Pentachlorophenol	87-86-5	D037	100 mg/L	2000 mg/kg
Pyridine	110-86-1	D038	5.0 mg/L	100 mg/kg
Tetrachloroethylene	127-18-4	D039	0.7 mg/L	14 mg/kg
Trichloroethylene	79-01-6	D040	0.5 mg/L	10 mg/kg

FIGURE 4, cont'd

CHARACTERISTIC TOXIC HAZARDOUS WASTE AND TCLP CONCENTRATIONS

Chemical Name	CAS Number	Waste Code	TCLP conc. liquid	Equivalent conc. In Soil
2,4,5-Trichlorophenol	95-95-4	D041	400 mg/L	8000 mg/kg
2,4,6-Trichlorophenol	88-06-2	D042	2.0 mg/L	40 mg/kg
Vinyl Chloride	75-01-4	D043	0.2 mg/L	4.0 mg/kg

TITLE: SAMPLE DISPOSAL

FIGURE 5

EXAMPLE OF ELEMENTARY NEUTRALIZATION LOGBOOK

Katahdin Analytical Services, Inc. – Elementary Neutralization Logbook

Date: 3-4-09		**Time:** 12:00	**Analyst:** GN
# of gallons neutralized	Final pH	Condition of drain and sink area before and after neutralization.	Significant Repairs or Corrective Actions
5	5	good	
6	7	good	
6	5	good	
6	6	good	
2	8	good	

25
240

Date: 3-10-09		**Time:** 13:45	**Analyst:** GN
# of gallons neutralized	Final pH	Condition of drain and sink area before and after neutralization.	Significant Repairs or Corrective Actions
6	7	good	
6	6	good	
5	6	good	
6	8	good	
6	5	good	
6	8	good	
5	5	good	
6	7	good	
3	5	good	

49
289

Mobile Laboratory
Matrix: Groundwater
Analytical Group: VOCs

Analyte	CAS Number	Project Action Limit (µg/L)	Project Action Limit Reference	Project Quantitation Limit Goal (µg/L)	KB Labs		
					LOQ (µg/L)	LOD (µg/L)	MDL (µg/L)
cis-1,2-Dichloroethene	156-59-2	70	FDEP Residential GCTL	23	1.0	1.0	0.28
trans-1,2-Dichloroethene	156-60-5	100	FDEP Residential GCTL	33	1.0	1.0	0.20
Isopropyl benzene	98-82-8	0.8	FDEP Residential GCTL	0.27	0.5	0.5	0.19
Tetrachloroethene	127-18-4	3	FDEP Residential GCTL	1.0	1.0	1.0	0.18
Trichloroethene	79-01-6	3	FDEP Residential GCTL	1.0	1.0	1.0	0.26
1,1,1-Trichloroethane	71-55-6	200	FDEP Residential GCTL	67	1.0	1.0	0.49
1,1-Dichloroethane	75-34-3	70	FDEP Residential GCTL	23	1.0	1.0	0.40
1,1-Dichloroethene	75-35-4	7	FDEP Residential GCTL	2.3	1.0	1.0	0.45
1,2-Dichloroethane	107-06-2	3	FDEP Residential GCTL	1.0	1.0	1.0	0.20
Chloroethane	75-00-3	12	FDEP Residential GCTL	4.0	1.0	1.0	0.37
Toluene	108-88-3	40	FDEP Residential GCTL	13	1.0	1.0	0.17
Vinyl Chloride	75-01-4	1	FDEP Residential GCTL	0.33	1.0	1.0	0.21

CAS = Chemical Abstract Service
LOQ = Limit of Quantitation
LOD = Limit of Detection
ug/L = Micrograms per liter

LABORATORY ACCREDITATION BUREAU

Certificate Number L2223

Certificate of Accreditation

Accredited to DoD ELAP and ISO/IEC 17025:2005

Katahdin Analytical Services, Inc.

600 Technology Way
Scarborough, ME 04074

has met the requirements set forth in L-A-B's policies and procedures, all requirements of ISO/IEC 17025:2005 "General Requirements for the competence of Testing and Calibration Laboratories" and the U.S. Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP).*

The accredited lab has demonstrated technical competence to a defined "Scope of Accreditation" and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated 8 January 2009).

Accreditation Granted through: November 4, 2012

R. Douglas Leonard, Jr., Managing Director
Laboratory Accreditation Bureau
Presented the 4th of November, 2009

*See the laboratory's Scope of Accreditation for details of the DoD ELAP requirements
Laboratory Accreditation Bureau is found to be in compliance with ISO/IEC 17011:2004 and recognized by ILAC (International Laboratory Accreditation Cooperation) and NACLA (National Cooperation for Laboratory Accreditation).

LABORATORY ACCREDITATION BUREAU

Certificate # L2223

Scope of Accreditation For Katahdin Analytical Services

600 Technology Way
Scarborough, ME 04074
Leslie Dimond
1- 207-874-2400

In recognition of a successful assessment to ISO/IEC 17025:2005 and the requirements of the DoD Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in the DoD Quality Systems Manual for Environmental Laboratories (DoD QSM v4.1) based on the National Environmental Laboratory Accreditation Conference Chapter 5 Quality Systems Standard (NELAC Voted Revision June 5, 2003), accreditation is granted to Katahdin Analytical Services to perform the following tests:

Accreditation granted through: **November 4, 2012**

Testing - Environmental

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/ECD	608 / 8081A,B/ SOM01.2	4 4`-DDD
GC/ECD	608 / 8081A,B/ SOM01.2	4 4`-DDE
GC/ECD	608 / 8081A,B / SOM01.2	4 4`-DDT
GC/ECD	608 / 8081A,B / SOM01.2	Aldrin
GC/ECD	608 / 8081A,B / SOM01.2	alpha-BHC (alpha-Hexachlorocyclohexane)
GC/ECD	8081A,B / SOM01.2	Alpha-Chlordane
GC/ECD	608 / 8081A,B / SOM01.2	beta-BHC (beta-Hexachlorocyclohexane)
GC/ECD	608 /8081A,B	Chlordane (tech.)
GC/ECD	608 / 8081A,B / SOM01.2	delta-BHC
GC/ECD	608 / 8081A,B / SOM01.2	Dieldrin
GC/ECD	608 / 8081A,B / SOM01.2	Endosulfan I
GC/ECD	608 / 8081A,B / SOM01.2	Endosulfan II
GC/ECD	608 / 8081A,B / SOM01.2	Endosulfan sulfate
GC/ECD	608 / 8081A,B / SOM01.2	Endrin
GC/ECD	608 / 8081A,B / SOM01.2	Endrin aldehyde
GC/ECD	8081A,B / SOM01.2	Endrin Ketone
GC/ECD	8081A,B / SOM01.2	gamma-BHC (Lindane gamma-Hexachlorocyclohexane)
GC/ECD	608 / 8081A,B / SOM01.2	Heptachlor
GC/ECD	608 / 8081A,B / SOM01.2	Heptachlor epoxide

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/ECD	8081A,B / SOM01.2	Methoxychlor
GC/ECD	608 / 8081A,B / SOM01.2	Toxaphene (Chlorinated camphene)
GC/ECD	608 / 8082/8082A / SOM01.2	Aroclor-1221 (PCB-1221)
GC/ECD	608 / 8082/8082A / SOM01.2	Aroclor-1232 (PCB-1232)
GC/ECD	608 / 8082/8082A / SOM01.2	Aroclor-1242 (PCB-1242)
GC/ECD	608 / 8082/8082A / SOM01.2	Aroclor-1248 (PCB-1248)
GC/ECD	608 / 8082/8082A / SOM01.2	Aroclor-1254 (PCB-1254)
GC/ECD	608 / 8082/8082A / SOM01.2	Aroclor-1260 (PCB-1260)
GC/ECD	8082/8082A	Aroclor-1262 (PCB-1262)
GC/ECD	8082/8082A	Aroclor-1268 (PCB-1268)
GC/ECD	8082/8082A	2 2' 3 3' 4 4' 5 5' 6-Nonachlorobiphenyl (BZ 206)
GC/ECD	8082/8082A	2 2' 3 3' 4 4' 5 6-Octachlorobiphenyl (BZ 195)
GC/ECD	8082/8082A	2 2' 3 3' 4 4' 5-Heptachlorobiphenyl (BZ 170)
GC/ECD	8082/8082A	2 2' 3 3' 4 4'-Hexachlorobiphenyl (BZ 128)
GC/ECD	8082/8082A	2 2' 3 4 4' 5 5'-Heptachlorobiphenyl (BZ 180)
GC/ECD	8082/8082A	2 2' 3 4 4' 5' 6-Heptachlorobiphenyl (BZ 183)
GC/ECD	8082/8082A	2 2' 3 4 4' 5'-Hexachlorobiphenyl (BZ 138)
GC/ECD	8082/8082A	2 2' 3 4 4' 6 6'-Heptachlorobiphenyl (BZ 184)
GC/ECD	8082/8082A	2 2' 3 4' 5 5' 6-Heptachlorobiphenyl (BZ 187)
GC/ECD	8082/8082A	2 2' 3 4 5'-Pentachlorobiphenyl (BZ 87)
GC/ECD	8082/8082A	2 2' 3 5'-Tetrachlorobiphenyl (BZ 44)
GC/ECD	8082/8082A	2 2' 4 4' 5 5'-Hexachlorobiphenyl (BZ 153)
GC/ECD	8082/8082A	2 2' 4 5 5'-Pentachlorobiphenyl (BZ 101)
GC/ECD	8082/8082A	2 2' 4' 5-Tetrachlorobiphenyl (BZ 49)
GC/ECD	8082/8082A	2 2' 5 5'-Tetrachlorobiphenyl (BZ 52)
GC/ECD	8082/8082A	2 2' 5-Trichlorobiphenyl (BZ 18)
GC/ECD	8082/8082A	2 3 3' 4 4' 5-Hexachlorobiphenyl (BZ 156)
GC/ECD	8082/8082A	2 3 3' 4 4' 5'-Hexachlorobiphenyl (BZ 157)
GC/ECD	8082/8082A	2 3 3' 4 4'-Pentachlorobiphenyl (BZ 105)
GC/ECD	8082/8082A	2 3 3’ 4 4’ 5 5’-Heptachlorobiphenyl (BZ 189)
GC/ECD	8082/8082A	2 3' 4 4' 5 5'-Hexachlorobiphenyl (BZ 167)
GC/ECD	8082/8082A	2 3' 4 4' 5-Pentachlorobiphenyl (BZ 118)
GC/ECD	8082/8082A	2 3' 4 4'5-Pentachlorobiphenyl (BZ 123)
GC/ECD	8082/8082A	2 3' 4 4'-Tetrachlorobiphenyl (BZ 66)
GC/ECD	8082/8082A	2 3’ 4 4’ 5-Pentachlorobiphenyl (BZ 114)
GC/ECD	8082/8082A	2 4 4'-Trichlorobiphenyl (BZ 28)
GC/ECD	8082/8082A	2 4'-Dichlorobiphenyl (BZ 8)
GC/ECD	8082/8082A	3 3' 4 4' 5 5'-Hexachlorobiphenyl (BZ 169)
GC/ECD	8082/8082A	3 3' 4 4' 5-Pentachlorobiphenyl (BZ 126)
GC/ECD	8082/8082A	3 3' 4 4'-Tetrachlorobiphenyl (BZ 77)
GC/ECD	8082/8082A	3 4 4’ 5-Tetrachlorobiphenyl (BZ 81)
GC/ECD	8082/8082A	Decachlorobiphenyl (BZ 209)
GC/ECD	8151A	2 4 5-T
GC/ECD	8151A	2 4-D
GC/ECD	8151A	2 4-DB

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/ECD	8151A	Dalapon
GC/ECD	8151A	Dicamba
GC/ECD	8151A	Dichloroprop
GC/ECD	8151A	DInoseb
GC/ECD	8151A	MCPA
GC/ECD	8151A	MCPP
GC/ECD	8151A	Pentachlorophenol
GC/ECD	8151A	Silvex (2 4 5-TP)
GC/FID	8015B/C	Diesel range organics (DRO)
GC/FID	8015B/C	Gasoline range organics (GRO)
GC/FID	8011 / 504	1 2-Dibromoethane (EDB)
GC/FID	8011 / 504	1 2-Dibromo-3-chloropropane
GC/FID	RSK-175	Methane Ethane Ethene
GC/MS	8260B,C / 524.2	1 1 1 2-Tetrachloroethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 1 1-Trichloroethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 1 2 2-Tetrachloroethane
GC/MS	SOM01.2	1 1 2-Trichloro-1 2 2-trifluoroethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 1 2-Trichloroethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 1-Dichloroethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 1-Dichloroethene
GC/MS	8260B,C / 524.2	1 1-Dichloropropene
GC/MS	8260B,C / SOM01.2 / 524.2	1 2 3-Trichlorobenzene
GC/MS	8260B,C / 524.2	1 2 3-Trichloropropane
GC/MS	8260B,C / SOM01.2 / 524.2	1 2 4-Trichlorobenzene
GC/MS	8260B,C / 524.2	1 2 4-Trimethylbenzene
GC/MS	8260B,C / SOM01.2 / 524.2	1 2-Dibromo-3-chloropropane
GC/MS	8260B,C / SOM01.2 / 524.2	1 2-Dibromoethane (EDB)
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 2-Dichlorobenzene
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 2-Dichloroethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 2-Dichloropropane
GC/MS	8260B,C / 524.2	1 3 5-Trimethylbenzene
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 3-Dichlorobenzene
GC/MS	8260B,C / 524.2	1 3-Dichloropropane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 4-Dichlorobenzene
GC/MS	8260B,C / SOM01.2	1 4-Dioxane
GC/MS	8260B,C / 524.2	2 2-Dichloropropane

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	8260B,C / SOM01.2 / 524.2	2-Butanone
GC/MS	624 / 8260B,C	2-Chloroethyl vinyl ether
GC/MS	8260B,C / 524.2	2-Chlorotoluene
GC/MS	8260B,C / SOM01.2 / 524.2	2-Hexanone
GC/MS	8260B,C / 524.2	4-Chlorotoluene
GC/MS	8260B,C / SOM01.2 / 524.2	4-Methyl-2-pentanone
GC/MS	8260B,C / SOM01.2 / 524.2	Acetone
GC/MS	8260B,C	Acetonitrile
GC/MS	624 / 8260B,C	Acrolein
GC/MS	624 / 8260B,C / 524.2	Acrylonitrile
GC/MS	8260B,C / 524.2	Allyl chloride
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Benzene
GC/MS	8260B,C / 524.2	Bromobenzene
GC/MS	8260B,C / SOM01.2 / 524.2	Bromochloromethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Bromodichloromethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Bromoform
GC/MS	8260B,C / SOM01.2 / 524.2	Carbon disulfide
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Carbon tetrachloride
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Chlorobenzene
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Chloroethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Chloroform
GC/MS	8260B,C	Chloroprene
GC/MS	8260B,C / SOM01.2 / 524.2	cis-1 2-Dichloroethene
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	cis-1 3-Dichloropropene
GC/MS	SOM01.2	Cyclohexane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Dibromochloromethane
GC/MS	8260B,C / 524.2	Dibromomethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Dichlorodifluoromethane
GC/MS	8260B,C / 524.2	Diethyl ether
GC/MS	8260B,C	Di-isopropylether
GC/MS	8260B,C / 524.2	Ethyl methacrylate
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Ethylbenzene
GC/MS	8260B,C	Ethyl-t-butylether
GC/MS	8260B,C / 524.2	Hexachlorobutadiene
GC/MS	8260B,C	Iodomethane
GC/MS	8260B,C	Isobutyl alcohol

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	8260B,C / SOM01.2 / 524.2	Isopropyl benzene
GC/MS	8260B,C / SOM01.2 / 524.2	m p-xylenes
GC/MS	8260B,C / 524.2	Methacrylonitrile
GC/MS	SOM01.2	Methyl acetate
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Methyl bromide (Bromomethane)
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Methyl chloride (Chloromethane)
GC/MS	8260B,C / 524.2	Methyl methacrylate
GC/MS	8260B,C / SOM01.2 / 524.2	Methyl tert-butyl ether
GC/MS	SOM01.2	Methylcyclohexane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Methylene chloride
GC/MS	8260B,C / 524.2	Naphthalene
GC/MS	8260B,C / 524.2	n-Butylbenzene
Gc/ms	8260B,C / 524.2	n-Propylbenzene
GC/MS	8260B,C / SOM01.2 / 524.2	o-Xylene
GC/MS	8260B,C / 524.2	p-Isopropyltoluene
GC/MS	8260B,C / 524.2	Propionitrile
GC/MS	8260B,C / 524.2	sec-butylbenzene
GC/MS	8260B,C / SOM01.2 / 524.2	Styrene
GC/MS	8260B,C	t-Amylmethylether
GC/MS	8260B,C / 524.2	tert-Butyl alcohol
GC/MS	8260B,C	tert-Butylbenzene
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Tetrachloroethene (Perchloroethylene)
GC/MS	8260B,C / 524.2	Tetrahydrofuran
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Toluene
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	trans-1 2-Dichloroethylene
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	trans-1 3-Dichloropropylene
GC/MS	8260B,C / 524.2	trans-1 4-Dichloro-2-butuene
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Trichloroethene (Trichloroethylene)
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Trichlorofluoromethane
GC/MS	8260B,C	Vinyl acetate
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Vinyl chloride
GC/MS	624 8260B,C	Xylene
GC/MS	8270C,D / SOM01.2	1 2 4 5-Tetrachlorobenzene
GC/MS	625 / 8270C,D / SOM01.2	1 2 4-Trichlorobenzene
GC/MS	625 / 8270C,D / SOM01.2	1 2-Dichlorobenzene
GC/MS	8270C,D	1 2-Diphenylhydrazine

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	8270C,D	1 3 5-Trinitrobenzene
GC/MS	625 / 8270C,D / SOM01.2	1 3-Dichlorobenzene
GC/MS	8270C,D	1 3-Dinitrobenzene
GC/MS	625 / 8270C,D / SOM01.2	1 4-Dichlorobenzene
GC/MS	8270C,D	1 4-Dioxane
GC/MS	8270C,D	1 4-Naphthoquinone
GC/MS	8270C,D	1 4-Phenylenediamine
GC/MS	8270C,D	1-Naphthylamine
GC/MS	8270C,D / SOM01.2	2 3 4 6-Tetrachlorophenol
GC/MS	8270C,D / SOM01.2	2 4 5-Trochlorophenol
GC/MS	625 / 8270C,D / SOM01.2	2 4 6-Trichlorophenol
GC/MS	625 / 8270C,D / SOM01.2	2 4-Dichlorophenol
GC/MS	625 / 8270C,D / SOM01.2	2 4-Dimethylphenol
GC/MS	625 / 8270C,D / SOM01.2	2 4-Dinitrophenol
GC/MS	625 / 8270C,D / SOM01.2	2 4-Dinitrotoluene (2 4-DNT)
GC/MS	8270C,D	2 6-Dichlorophenol
GC/MS	625 / 8270C,D / SOM01.2	2 6-Dinitrotoluene (2 6-DNT)
GC/MS	8270C,D	2-Acetylaminofluorene
GC/MS	625 / 8270C,D / SOM01.2	2-Chloronaphthalene
GC/MS	625 / 8270C,D / SOM01.2	2-Chlorophenol
GC/MS	625 / 8270C,D / SOM01.2	2-Methyl-4 6-dinitrophenol
GC/MS	8270C,D / SOM01.2	2-Methylnaphthalene
GC/MS	8270C,D / SOM01.2	2-Methylphenol
GC/MS	8270C,D	2-Naphthylamine
GC/MS	8270C,D	2-Nitroaniline
GC/MS	625 / 8270C,D / SOM01.2	2-Nitrophenol
GC/MS	8270C,D	2-Picoline
GC/MS	625 / 8270C,D / SOM01.2	3 3'-Dichlorobenzidine
GC/MS	8270C,D	3 3'-Dimethylbenzidine
GC/MS	8270C,D	3-Methylcholanthrene
GC/MS	8270C,D / SOM01.2	3-Nitroaniline
GC/MS	8270C,D	4-Aminobiphenyl
GC/MS	625 / 8270C,D / SOM01.2	4-Bromophenyl phenyl ether
GC/MS	625 / 8270C,D / SOM01.2	4-Chloro-3-methylphenol
GC/MS	8270C,D / SOM01.2	4-Chloroaniline
GC/MS	625 / 8270C,D / SOM01.2	4-Chlorophenyl phenylether
GC/MS	8270C,D	4-Dimethyl aminoazobenzene
GC/MS	8270C,D / SOM01.2	4-Methylphenol
GC/MS	8270C,D / SOM01.2	4-Nitroaniline
GC/MS	625 / 8270C,D / SOM01.2	4-Nitrophenol
GC/MS	8270C,D	5-Nitro-o-toluidine
GC/MS	8270C,D	7,12-Dimethylphenethylamine
GC/MS	8270C,D	a a-Dimethylphenethylamine
GC/MS	625 / 8270C,D / SOM01.2	Acenaphthene
GC/MS	625 / 8270C,D / SOM01.2	Acenaphthylene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	8270C,D / SOM01.2	Acetophenone
GC/MS	8270C,D	Aniline
GC/MS	625 / 8270C,D / SOM01.2	Anthracene
GC/MS	8270C,D	Aramite
GC/MS	8270C,D / SOM01.2	Atrazine
GC/MS	SOM01.2	Benzaldehyde
GC/MS	625 / 8270C,D	Benzidine
GC/MS	625 / 8270C,D / SOM01.2	Benzo(a)anthracene
GC/MS	625 / 8270C,D / SOM01.2	Benzo(a)pyrene
GC/MS	625 / 8270C,D / SOM01.2	Benzo(b)fluoranthene
GC/MS	625 / 8270C,D / SOM01.2	Benzo(g h i)perylene
GC/MS	625 / 8270C,D / SOM01.2	Benzo(k)fluoranthene
GC/MS	8270C,D	Benzoic Acid
GC/MS	8270C,D	Benzyl alcohol
GC/MS	8270C,D / SOM01.2	Biphenyl
GC/MS	625 / 8270C,D / SOM01.2	bis(2-Chloroethoxy)methane
GC/MS	625 / 8270C,D / SOM01.2	bis(2-Chloroethyl) ether
GC/MS	625 / 8270C,D / SOM01.2	bis(2-Chloroisopropyl) ether (2 2`-Oxybis(1-chloropropane))
GC/MS	625 / 8270C,D / SOM01.2	bis(2-Ethylhexyl) phthalate (DEHP)
GC/MS	625 / 8270C,D / SOM01.2	Butyl benzyl phthalate
GC/MS	SOM01.2	Caprolactam
GC/MS	8270C,D / SOM01.2	Carbazole
GC/MS	8270C,D	Chlorobenzilate
GC/MS	625 / 8270C,D / SOM01.2	Chrysene
GC/MS	8270C,D	Diallate
GC/MS	625 / 8270C,D / SOM01.2	Dibenz(a h)anthracene
GC/MS	8270C,D / SOM01.2	Dibenzofuran
GC/MS	625 / 8270C,D / SOM01.2	Diethyl phthalate
GC/MS	8270C,D	Dimethoate
GC/MS	625 / 8270C,D / SOM01.2	Dimethyl phthalate
GC/MS	625 / 8270C,D / SOM01.2	Di-n-butyl phthalate
GC/MS	625 / 8270C,D / SOM01.2	Di-n-octyl phthalate
GC/MS	8270C,D	Ethyl methanesulfonate
GC/MS	8270C,D	Famfur
GC/MS	625 / 8270C,D / SOM01.2	Fluoranthene
GC/MS	625 / 8270C,D / SOM01.2	Fluorene
GC/MS	625 / 8270C,D / SOM01.2	Hexachlorobenzene
GC/MS	625 / 8270C,D / SOM01.2	Hexachlorobutadiene
GC/MS	625 / 8270C,D / SOM01.2	Hexachlorocyclopentadiene
GC/MS	625 / 8270C,D / SOM01.2	Hexachloroethane
GC/MS	8270C,D	Hexachloropropene
GC/MS	625 / 8270C,D / SOM01.2	Indeno(1 2 3-cd)pyrene
GC/MS	8270C,D	Isodrin
GC/MS	625 / 8270C,D / SOM01.2	Isophorone

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	8270C,D	Isosafrole
GC/MS	8270C,D	Methapyriline
GC/MS	8270C,D	Methy methanesulfonate
GC/MS	8270C,D	Methyl parathion
GC/MS	625 / 8270C,D / SOM01.2	Naphthalene
GC/MS	625 / 8270C,D / SOM01.2	Nitrobenzene
GC/MS	8270C,D	Nitroquinoline-1-oxide
GC/MS	8270C,D	n-Nitrosodiethylamine
GC/MS	625 / 8270C,D / SOM01.2	n-Nitrosodimethylamine
GC/MS	8270C,D	n-Nitroso-di-n-butylamine
GC/MS	625 / 8270C,D / SOM01.2	n-Nitrosodi-n-propylamine
GC/MS	625 / 8270C,D / SOM01.2	n-Nitrosodiphenylamine
GC/MS	8270C,D	n-Nitrosomethylethylamine
GC/MS	8270C,D	n-Nitrosomorpholine
GC/MS	8270C,D	n-Nitrosopiperidine
GC/MS	8270C,D	n-Nitrosopyrrolidine
GC/MS	8270C,D	o o o-Triethyl phosphorothioate
GC/MS	8270C,D	o-Toluidine
GC/MS	8270C,D	Pentachlorobenzene
GC/MS	8270C,D	Pentachloronitrobenzene
GC/MS	625 / 8270C,D / SOM01.2	Pentachlorophenol
GC/MS	8270C,D	Phenacetin
GC/MS	625 / 8270C,D / SOM01.2	Phenanthrene
GC/MS	625 / 8270C,D / SOM01.2	Phenol
GC/MS	8270C,D	Phorate
GC/MS	8270C,D	Pronamide
GC/MS	625 / 8270C,D / SOM01.2	Pyrene
GC/MS	8270C,D	Pyrididne
GC/MS	8270C,D	Safrole
GC/MS	8270C,D	Thionazin
HPLC	8330/8330A/8330B	1 3 5-Trinitrobenzene
HPLC	8330/8330A/8330B	1 3-Dinitrobenzene
HPLC	8330/8330A/8330B	2 4 6-Trinitrotoluene
HPLC	8330/8330A/8330B	2 4-Dinitrotoluene
HPLC	8330/8330A/8330B	2 6-Dinitrotoluene
HPLC	8330/8330A/8330B	2-Amino-4 6 -dinitrotoluene
HPLC	8330/8330A/8330B	2-Nitrotoluene
HPLC	8330/8330A/8330B	3-Nitrotoluene
HPLC	8330/8330A/8330B	4-Amino-2,3-dinitrotoluene
HPLC	8330/8330A/8330B	4-Nitrotoluene
HPLC	8330/8330A/8330B	Hexahydro-1 3 5-trinitro-1 3 5-triazine (RDX)
HPLC	8330/8330A/8330B	Nitrobenzene
HPLC	8330/8330A/8330B	Nitroglycerin
HPLC	8330/8330A/8330B	Octahydro-1 3 5 7-tetrazocine (HMX)
HPLC	8330/8330A/8330B	Tetryl

Non-Potable Water		
Technology	**Method**	**Analyte**
CVAA	245.1 / 7470A / ILM05.3	Mercury
CVAF	1631E	Low Level Mercury
ICP	200.7 / 6010B,C / ILM05.3	Aluminum
ICP	200.7 / 6010B,C / ILM05.3	Antimony
ICP	200.7 / 6010B,C / ILM05.3	Arsenic
ICP	200.7 / 6010B,C / ILM05.3	Barium
ICP	200.7 / 6010B,C / ILM05.3	Beryllium
ICP	200.7 / 6010B,C	Boron
ICP	200.7 / 6010B,C / ILM05.3	Cadmium
ICP	200.7 / 6010B,C / ILM05.3	Calcium
ICP	200.7 / 6010B,C / ILM05.3	Chromium
ICP	200.7 / 6010B,C / ILM05.3	Cobalt
ICP	200.7 / 6010B,C / ILM05.3	Copper
ICP	200.7 / 6010B,C / ILM05.3	Iron
ICP	200.7 / 6010B,C / ILM05.3	Lead
ICP	200.7 / 6010B,C / ILM05.3	Magnesium
ICP	200.7 / 6010B,C / ILM05.3	Manganese
ICP	200.7 / 6010B,C	Molybdenum
ICP	200.7 / 6010B,C / ILM05.3	Nickel
ICP	200.7 / 6010B,C / ILM05.3	Potassium
ICP	200.7 / 6010B,C / ILM05.3	Selenium
ICP	200.7	Silicon
ICP	200.7 / 6010B,C / ILM05.3	Silver
ICP	200.7 / 6010B,C / ILM05.3	Sodium
ICP	6010B,C	Strontium
ICP	200.7 / 6010B,C / ILM05.3	Thallium
ICP	200.7 / 6010B,C	Tin
ICP	200.7 / 6010B,C	Titanium
ICP	200.7 / 6010B,C / ILM05.3	Vanadium
ICP	200.7 / 6010B,C / ILM05.3	Zinc
ICP/MS	200.8 / 6020/6020A / ILM05.3	Aluminum
ICP/MS	200.8 / 6020/6020A / ILM05.3	Antimony
ICP/MS	200.8 / 6020/6020A / ILM05.3	Arsenic
ICP/MS	200.8 / 6020/6020A / ILM05.3	Barium
ICP/MS	200.8 / 6020/6020A / ILM05.3	Beryllium
ICP/MS	200.8 / 6020/6020A	Boron
ICP/MS	200.8 / 6020/6020A / ILM05.3	Cadmium
ICP/MS	200.8 / 6020/6020A	Calcium
ICP/MS	200.8 / 6020/6020A / ILM05.3	Chromium
ICP/MS	200.8 / 6020/6020A / ILM05.3	Cobalt
ICP/MS	200.8 / 6020/6020A / ILM05.3	Copper
ICP/MS	200.8 / 6020/6020A	Iron
ICP/MS	200.8 / 6020/6020A / ILM05.3	Lead
ICP/MS	200.8 / 6020/6020A / ILM05.3	Magnesium
ICP/MS	200.8 / 6020/6020A	Manganese

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP/MS	200.8 / 6020/6020A	Molybdenum
ICP/MS	200.8 / 6020/6020A / ILM05.3	Nickel
ICP/MS	200.8 / 6020/6020A	Potassium
ICP/MS	200.8 / 6020/6020A / ILM05.3	Selenium
ICP/MS	200.8 / 6020/6020A / ILM05.3	Silicon
ICP/MS	200.8 / 6020/6020A / ILM05.3	Silver
ICP/MS	200.8 / 6020/6020A	Sodium
ICP/MS	6020/6020A	Strontium
ICP/MS	200.8 / 6020/6020A / ILM05.3	Thallium
ICP/MS	200.8 / 6020/6020A	Tin
ICP/MS	200.8 / 6020/6020A	Titanium
ICP/MS	200.8	Uranium
ICP/MS	200.8 / 6020/6020A / ILM05.3	Vanadium
ICP/MS	200.8 / 6020/6020A / ILM05.3	Zinc
IC	300.0 / 9056/9056A	Bromide
IC	300.0 / 9056/9056A	Chloride
IC	300.0 / 9056/9056A	Nitrate as N
IC	300.0 / 9056/9056A	Nitrite as N
IC	300.0 / 9056/9056A	Nitrate + Nitrite
IC	300.0 / 9056/9056A	Orthophosphate as P
IC	300.0 / 9056/9056A	Sulfate
Titration	310.2 / 2320B	Alkalinity
Caculation	2340C	Hardness
Gravimetric	1664A	Oil and Grease
Gravimetric	2540 B, C, D	Solids
ISE	120.1 / 2510 B	Conductivity
ISE	2520B	Practical Salinity
ISE	4500F- C	Fluoride
ISE	4500H+ B	pH
ISE	5210B	TBOD / CBOD
Physical	1010 A	Ignitability
Physical	9040C	pH
Titration	2340B	Hardness
Titration	4500SO_3 B	Sulfite
Titration	9034 / 4500S^{2-} E	Sulfide
Titration	Chap. 7.3.4	Reactive Sulfide
TOC	9060A / 5310B	Total organic carbon
Turbidimetric	180.1 / 2130B	Turbidity
Turbidimetric	9038 / ASTM 516-02	Sulfate
UV/VIS	335.4 / 9012B / 4500-CN G	Amenable cyanide
UV/VIS	350.1 / 4500NH3 H	Ammonia as N
UV/VIS	3500Fe D	Ferrous Iron
UV/VIS	351.2	Kjeldahl nitrogen - total
UV/VIS	353.2 / 4500NO3 F	Nitrate + Nitrite

Non-Potable Water		
Technology	**Method**	**Analyte**
UV/VIS	353.2 / 4500NO3 F	Nitrate as N
UV/VIS	353.2 / 4500NO3 F	Nitrite as N
UV/VIS	365.1 / 4500P E	Orthophosphate as P
UV/VIS	365.4	Phosphorus total
UV/VIS	376.3	AVS-SEM
UV/VIS	410.4	COD
UV/VIS	420.1 / 9065	Total Phenolics
UV/VIS	4500Cl G	Total Residual Chlorine
UV/VIS	5540C	MBAS
UV/VIS	7196A / 3500-Cr D	Chromium VI
UV/VIS	9012B / ILM05.3/ 335.4	Total Cyanide
UV/VIS	9251 / 4500Cl E	Chloride
UV/VIS	Chap. 7.3.4	Reactive Cyanide
Preparation	**Method**	**Type**
Cleanup Methods	3640A	Gel Permeation Clean-up
Cleanup Methods	3630C	Silica Gel
Cleanup Methods	3660B	Sulfur Clean-Up
Cleanup Methods	3665A	Sulfuric Acid Clean-Up
Organic Preparation	3510C	Separatory Funnel Extraction
Organic Preparation	3520C	Continuous Liquid-Liquid Extraction
Inorganic Preparation	3010A	Hotblock
Volatile Organic Preparation	5030B,C	Purge and Trap

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/ECD	8081A,B/ SOM01.2	4 4`-DDD
GC/ECD	8081A,B / SOM01.2	4 4`-DDE
GC/ECD	8081A,B / SOM01.2	4 4`-DDT
GC/ECD	8081A,B / SOM01.2	Aldrin
GC/ECD	8081A,B / SOM01.2	alpha-BHC (alpha-Hexachlorocyclohexane)
GC/ECD	8081A,B / SOM01.2	Alpha-Chlordane
GC/ECD	8081A,B / SOM01.2	beta-BHC (beta-Hexachlorocyclohexane)
GC/ECD	608 /8081A,B	Chlordane (tech.)
GC/ECD	8081A,B / SOM01.2	delta-BHC
GC/ECD	8081A,B / SOM01.2	Dieldrin
GC/ECD	8081A,B / SOM01.2	Endosulfan I
GC/ECD	8081A,B / SOM01.2	Endosulfan II
GC/ECD	8081A,B / SOM01.2	Endosulfan sulfate
GC/ECD	8081A,B / SOM01.2	Endrin
GC/ECD	8081A,B / SOM01.2	Endrin aldehyde
GC/ECD	8081A,B / SOM01.2	Endrin Ketone

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/ECD	8081A,B / SOM01.2	gamma-BHC (Lindane gamma-Hexachlorocyclohexane)
GC/ECD	8081A,B / SOM01.2	Heptachlor
GC/ECD	8081A,B / SOM01.2	Heptachlor epoxide
GC/ECD	8081A,B / SOM01.2	Methoxychlor
GC/ECD	8081A,B / SOM01.2	Toxaphene (Chlorinated camphene)
GC/ECD	8082/8082A/ SOM01.2	Aroclor-1016 (PCB-1016)
GC/ECD	8082/8082A/ SOM01.2	Aroclor-1221 (PCB-1221)
GC/ECD	8082/8082A/ SOM01.2	Aroclor-1232 (PCB-1232)
GC/ECD	8082/8082A/ SOM01.2	Aroclor-1242 (PCB-1242)
GC/ECD	8082/8082A/ SOM01.2	Aroclor-1248 (PCB-1248)
GC/ECD	8082/8082A/ SOM01.2	Aroclor-1254 (PCB-1254)
GC/ECD	8082/8082A/ SOM01.2	Aroclor-1260 (PCB-1260)
GC/ECD	8082/8082A	Aroclor-1262 (PCB-1262)
GC/ECD	8082/8082A	Aroclor-1268 (PCB-1268)
GC/ECD	8082/8082A	2 2' 3 3' 4 4' 5 5' 6-Nonachlorobiphenyl (BZ 206)
GC/ECD	8082/8082A	2 2' 3 3' 4 4' 5 6-Octachlorobiphenyl (BZ 195)
GC/ECD	8082/8082A	2 2' 3 3' 4 4' 5-Heptachlorobiphenyl (BZ 170)
GC/ECD	8082/8082A	2 2' 3 3' 4 4'-Hexachlorobiphenyl (BZ 128)
GC/ECD	8082/8082A	2 2' 3 4 4' 5 5'-Heptachlorobiphenyl (BZ 180)
GC/ECD	8082/8082A	2 2' 3 4 4' 5' 6-Heptachlorobiphenyl (BZ 183)
GC/ECD	8082/8082A	2 2' 3 4 4' 5'-Hexachlorobiphenyl (BZ 138)
GC/ECD	8082/8082A	2 2' 3 4 4' 6 6'-Heptachlorobiphenyl (BZ 184)
GC/ECD	8082/8082A	2 2' 3 4' 5 5' 6-Heptachlorobiphenyl (BZ 187)
GC/ECD	8082/8082A	2 2' 3 4 5'-Pentachlorobiphenyl (BZ 87)
GC/ECD	8082/8082A	2 2' 3 5'-Tetrachlorobiphenyl (BZ 44)
GC/ECD	8082/8082A	2 2' 4 4' 5 5'-Hexachlorobiphenyl (BZ 153)
GC/ECD	8082/8082A	2 2' 4 5 5'-Pentachlorobiphenyl (BZ 101)
GC/ECD	8082/8082A	2 2' 4' 5-Tetrachlorobiphenyl (BZ 49)
GC/ECD	8082/8082A	2 2' 5 5'-Tetrachlorobiphenyl (BZ 52)
GC/ECD	8082/8082A	2 2' 5-Trichlorobiphenyl (BZ 18)
GC/ECD	8082/8082A	2 3 3' 4 4' 5-Hexachlorobiphenyl (BZ 156)
GC/ECD	8082/8082A	2 3 3' 4 4' 5'-Hexachlorobiphenyl (BZ 157)
GC/ECD	8082/8082A	2 3 3' 4 4'-Pentachlorobiphenyl (BZ 105)
GC/ECD	8082/8082A	2 3 3’ 4 4’ 5 5’-Heptachlorobiphenyl (BZ 189)
GC/ECD	8082/8082A	2 3' 4 4' 5 5'-Hexachlorobiphenyl (BZ 167)
GC/ECD	8082/8082A	2 3' 4 4' 5-Pentachlorobiphenyl (BZ 118)
GC/ECD	8082/8082A	2 3' 4 4'5-Pentachlorobiphenyl (BZ 123)
GC/ECD	8082/8082A	2 3' 4 4'-Tetrachlorobiphenyl (BZ 66)
GC/ECD	8082/8082A	2 3’ 4 4’ 5-Pentachlorobiphenyl (BZ 114)
GC/ECD	8082/8082A	2 4 4'-Trichlorobiphenyl (BZ 28)
GC/ECD	8082/8082A	2 4'-Dichlorobiphenyl (BZ 8)
GC/ECD	8082/8082A	3 3' 4 4' 5 5'-Hexachlorobiphenyl (BZ 169)
GC/ECD	8082/8082A	3 3' 4 4' 5-Pentachlorobiphenyl (BZ 126)
GC/ECD	8082/8082A	3 3' 4 4'-Tetrachlorobiphenyl (BZ 77)

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/ECD	8082/8082A	3 4 4' 5-Tetrachlorobiphenyl (BZ 81)
GC/ECD	8082/8082A	Decachlorobiphenyl (BZ 209)
GC/ECD	8151A	2 4 5-T
GC/ECD	8151A	2 4-D
GC/ECD	8151A	2 4-DB
GC/ECD	8151A	Dalapon
GC/ECD	8151A	Dicamba
GC/ECD	8151A	Dichloroprop
GC/ECD	8151A	DInoseb
GC/ECD	8151A	MCPA
GC/ECD	8151A	MCPP
GC/ECD	8151A	Pentachlorophenol
GC/ECD	8151A	Silvex (2 4 5-TP)
GC/FID	8015B,C	Diesel range organics (DRO)
GC/FID	8015B,C	Gasoline range organics (GRO)
GC/FID	8011	EDB
GC/FID	8011	1 2-Dibromo-3-chloropropane
GC/MS	8260B,C	1 1 1 2-Tetrachloroethane
GC/MS	8260B,C / SOM01.2	1 1 1-Trichloroethane
GC/MS	8260B,C / SOM01.2	1 1 2 2-Tetrachloroethane
GC/MS	SOM01.2	1 1 2-Trichloro-1 2 2-trifluoroethane
GC/MS	8260B,C / SOM01.2	1 1 2-Trichloroethane
GC/MS	8260B,C / SOM01.2	1 1-Dichloroethane
GC/MS	8260B,C / SOM01.2	1 1-Dichloroethylene
GC/MS	8260B,C	1 1-Dichloropropene
GC/MS	8260B,C / SOM01.2	1 2 3-Trichlorobenzene
GC/MS	8260B,C	1 2 3-Trichloropropane
GC/MS	8260B,C / SOM01.2	1 2 4-Trichlorobenzene
GC/MS	8260B,C	1 2 4-Trimethylbenzene
GC/MS	8260B,C / SOM01.2	1 2-Dibromo-3-chloropropane
GC/MS	8260B,C / SOM01.2	1 2-Dichlorobenzene
GC/MS	8260B,C / SOM01.2	1 2-Dichloroethane
GC/MS	8260B,C / SOM01.2	1 2-Dichloropropane
GC/MS	8260B,C	1 3 5-Trimethylbenzene
GC/MS	8260B,C / SOM01.2	1 3-Dichlorobenzene
GC/MS	8260B,C	1 3-Dichloropropane
GC/MS	8260B,C / SOM01.2	1 4-Dichlorobenzene
GC/MS	8260B,C / SOM01.2	1 4-Dioxane
GC/MS	8260B,C	2 2-Dichloropropane
GC/MS	8260B,C / SOM01.2	2-Butanone
GC/MS	8260B,C	2-Chloroethyl vinyl ether
GC/MS	8260B,C	2-Chlorotoluene
GC/MS	8260B,C / SOM01.2	2-Hexanone
GC/MS	8260B,C	4-Chlorotoluene
GC/MS	8260B,C / SOM01.2	4-Methyl-2-pentanone

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	8260B,C / SOM01.2	Acetone
GC/MS	8260B,C	Acetonitrile
GC/MS	8260B,C	Acrolein
GC/MS	8260B,C	Acrylonitrile
GC/MS	8260B,C	Allyl chloride
GC/MS	8260B,C / SOM01.2	Benzene
GC/MS	8260B,C	Bromobenzene
GC/MS	8260B,C / SOM01.2	Bromochloromethane
GC/MS	8260B,C / SOM01.2	Bromodichloromethane
GC/MS	8260B,C / SOM01.2	Bromoform
GC/MS	8260B,C / SOM01.2	Carbon disulfide
GC/MS	8260B,C / SOM01.2	Carbon tetrachloride
GC/MS	8260B,C / SOM01.2	Chlorobenzene
GC/MS	8260B,C / SOM01.2	Chloroethane
GC/MS	8260B,C / SOM01.2	Chloroform
GC/MS	8260B,C	Chloroprene
GC/MS	8260B,C / SOM01.2	cis-1 2-Dichloroethene
GC/MS	8260B,C / SOM01.2	cis-1 3-Dichloropropene
GC/MS	SOM01.2	Cyclohexane
GC/MS	8260B,C / SOM01.2	Dibromochloromethane
GC/MS	8260B,C	Dibromomethane
GC/MS	624 / 8260B,C / SOM01.2	Dichlorodifluoromethane
GC/MS	8260B,C	Diethyl ether
GC/MS	8260B,C	Di-isopropylether
GC/MS	8260B,C / SOM01.2	EDB
GC/MS	8260B,C	Ethyl methacrylate
GC/MS	8260B,C / SOM01.2	Ethylbenzene
GC/MS	8260B,C	Ethyl-t-butylether
GC/MS	8260B,C	Hexachlorobutadiene
GC/MS	8260B,C	Iodomethane
GC/MS	8260B,C	Isobutyl alcohol
GC/MS	8260B,C / SOM01.2	Isopropyl benzene
GC/MS	SOM01.2	m p-xylenes
GC/MS	8260B,C	Methacrylonitrile
GC/MS	SOM01.2	Methyl acetate
GC/MS	8260B,C / SOM01.2	Methyl bromide (Bromomethane)
GC/MS	8260B,C / SOM01.2	Methyl chloride (Chloromethane)
GC/MS	8260B,C	Methyl methacrylate
GC/MS	8260B,C / SOM01.2	Methyl tert-butyl ether
GC/MS	SOM01.2	Methylcyclohexane
GC/MS	8260B,C / SOM01.2	Methylene chloride
GC/MS	8260B,C	Naphthalene
GC/MS	8260B,C	n-Butylbenzene
GC/MS	8260B,C	n-proplybenzene
GC/MS	8260B,C	o-Xylene

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	8260B,C	p-Isopropyltoluene
GC/MS	8260B,C	Propionitrile
GC/MS	8260B,C	sec-butylbenzene
GC/MS	8260B,C / SOM01.2	Styrene
GC/MS	8260B,C	t-Amylmethylether
GC/MS	8260B,C	tert-Butyl alcohol
GC/MS	8260B,C	tert-Butylbenzene
GC/MS	8260B,C / SOM01.2	Tetrachloroethylene (Perchloroethylene)
GC/MS	8260B,C	Tetrahydrofuran
GC/MS	8260B,C / SOM01.2	Toluene
GC/MS	8260B,C / SOM01.2	trans-1 2-Dichloroethylene
GC/MS	8260B,C / SOM01.2	trans-1 3-Dichloropropylene
GC/MS	8260B,C	Trans-1 4-Dichloro-2-butuene
GC/MS	8260B,C / SOM01.2	Trichloroethene (Trichloroethylene)
GC/MS	8260B,C / SOM01.2	Trichlorofluoromethane
GC/MS	8260B,C	Vinyl acetate
GC/MS	8260B,C / SOM01.2	Vinyl chloride
GC/MS	8260B,C	Xylene
GC/MS	8270C,D	1-Naphthylamine
GC/MS	8270C,D	2-Acetylaminofluorene
GC/MS	8270C,D / SOM01.2	2-Chloronaphthalene
GC/MS	8270C,D / SOM01.2	2-Chlorophenol
GC/MS	8270C,D / SOM01.2	2-Methylnaphthalene
GC/MS	8270C,D / SOM01.2	2-Methylphenol
GC/MS	8270C,D	2-Naphthylamine
GC/MS	8270C,D	2-Nitroaniline
GC/MS	8270C,D / SOM01.2	2-Nitrophenol
GC/MS	8270C,D	2-Picoline
GC/MS	8270C,D	3-Methylcholanthrene
GC/MS	8270C,D / SOM01.2	3-Nitroaniline
GC/MS	8270C,D	4-Aminobiphenyl
GC/MS	8270C,D / SOM01.2	4-Bromophenyl phenyl ether
GC/MS	8270C,D / SOM01.2	4-Chloro-3-methylphenol
GC/MS	8270C,D / SOM01.2	4-Chloroaniline
GC/MS	8270C,D / SOM01.2	4-Chlorophenyl phenylether
GC/MS	8270C,D	4-Dimethyl aminoazobenzene
GC/MS	8270C,D / SOM01.2	4-Methylphenol
GC/MS	8270C,D / SOM01.2	4-Nitroaniline
GC/MS	8270C,D / SOM01.2	4-Nitrophenol
GC/MS	8270C,D	5-Nitro-o-toluidine
GC/MS	8270C,D	a a-Dimethylphenethylamine
GC/MS	8270C,D / SOM01.2	Acenaphthene
GC/MS	8270C,D / SOM01.2	Acenaphthylene
GC/MS	8270C,D / SOM01.2	Acetophenone
GC/MS	8270C,D	Aniline

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	8270C,D / SOM01.2	Anthracene
GC/MS	8270C,D	Aramite
GC/MS	8270C,D / SOM01.2	Atrazine
GC/MS	SOM01.2	Benzaldehyde
GC/MS	8270C,D	Benzidine
GC/MS	8270C,D / SOM01.2	Benzo(a)anthracene
GC/MS	8270C,D / SOM01.2	Benzo(a)pyrene
GC/MS	8270C,D / SOM01.2	Benzo(b)fluoranthene
GC/MS	8270C,D / SOM01.2	Benzo(g h i)perylene
GC/MS	8270C,D / SOM01.2	Benzo(k)fluoranthene
GC/MS	8270C,D	Benzoic Acid
GC/MS	8270C,D	Benzyl alcohol
GC/MS	8270C,D / SOM01.2	Biphenyl
GC/MS	8270C,D / SOM01.2	bis(2-Chloroethoxy)methane
GC/MS	8270C,D / SOM01.2	bis(2-Chloroethyl) ether
GC/MS	8270C,D / SOM01.2	bis(2-Ethylhexyl) phthalate (DEHP)
GC/MS	8270C,D / SOM01.2	Butyl benzyl phthalate
GC/MS	SOM01.2	Caprolactam
GC/MS	8270C,D / SOM01.2	Carbazole
GC/MS	8270C,D	Chlorobenzilate
GC/MS	8270C,D / SOM01.2	Chrysene
GC/MS	8270C,D	Diallate
GC/MS	8270C,D / SOM01.2	Dibenz(a h)anthracene
GC/MS	8270C,D / SOM01.2	Dibenzofuran
GC/MS	8270C,D / SOM01.2	Diethyl phthalate
GC/MS	8270C,D	Dimethoate
GC/MS	8270C,D / SOM01.2	Dimethyl phthalate
GC/MS	8270C,D / SOM01.2	Di-n-butyl phthalate
GC/MS	8270C,D / SOM01.2	Di-n-octyl phthalate
GC/MS	8270C,D	Ethyl methanesulfonate
GC/MS	8270C,D	Famfur
GC/MS	8270C,D / SOM01.2	Fluoranthene
GC/MS	8270C,D / SOM01.2	Fluorene
GC/MS	8270C,D / SOM01.2	Hexachlorobenzene
GC/MS	8270C,D / SOM01.2	Hexachlorobutadiene
GC/MS	8270C,D / SOM01.2	Hexachlorocyclopentadiene
GC/MS	8270C,D / SOM01.2	Hexachloroethane
GC/MS	8270C,D	Hexachloropropene
GC/MS	8270C,D	Isodrin
GC/MS	8270C,D / SOM01.2	Isophorone
GC/MS	8270C,D	Isosafrole
GC/MS	8270C,D	Methapyriline
GC/MS	8270C,D	Methyl methanesulfonate
GC/MS	8270C,D	Methyl parathion
GC/MS	8270C,D / SOM01.2	Naphthalene

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	8270C,D / SOM01.2	Nitrobenzene
GC/MS	8270C,D	Nitroquinoline-1-oxide
GC/MS	8270C,D	n-Nitrosodiethylamine
GC/MS	8270C,D / SOM01.2	n-Nitrosodimethylamine
GC/MS	8270C,D	n-Nitroso-di-n-butylamine
GC/MS	8270C,D / SOM01.2	n-Nitrosodi-n-propylamine
GC/MS	8270C,D / SOM01.2	n-Nitrosodiphenylamine
GC/MS	8270C,D	n-Nitrosomethylethylamine
GC/MS	8270C,D	n-Nitrosomorpholine
GC/MS	8270C,D	n-Nitrosopiperidine
GC/MS	8270C,D	n-Nitrosopyrrolidine
GC/MS	8270C,D	o o o-Triethyl phosphorothioate
GC/MS	8270C,D	o-Toluidine
GC/MS	8270C,D	Pentachlorobenzene
GC/MS	8270C,D	Pentachloronitrobenzene
GC/MS	8270C,D/ SOM01.2	Pentachlorophenol
GC/MS	8270C,D	Phenacetin
GC/MS	8270C,D / SOM01.2	Phenanthrene
GC/MS	8270C,D / SOM01.2	Phenol
GC/MS	8270C,D	Phorate
GC/MS	8270C,D	Pronamide
GC/MS	8270C,D / SOM01.2	Pyrene
GC/MS	8270C,D	Pyrididne
GC/MS	8270C,D	Safrole
GC/MS	8270C,D	Thionazin
GC/MS	8270C,D / SOM01.2	Indeno(1 2 3-cd)pyrene
GC/MS	8270C,D / SOM01.2	1 2 4-Trichlorobenzene
GC/MS	8270C,D	1 3 5-Trinitrobenzene
GC/MS	8270C,D / SOM01.2	1 2 4 5-Tetrachlorobenzene
GC/MS	8270C,D / SOM01.2	2 4 5-Trochlorophenol
GC/MS	8270C,D / SOM01.2	2 4 6-Trichlorophenol
GC/MS	8270C,D / SOM01.2	2 3 4 6-Tetrachlorophenol
GC/MS	8270C,D / SOM01.2	1 2-Dichlorobenzene
GC/MS	8270C,D	1 2-Diphenylhydrazine
GC/MS	8270C,D / SOM01.2	1 3-Dichlorobenzene
GC/MS	8270C,D	1 3-Dinitrobenzene
GC/MS	8270C,D / SOM01.2	1 4-Dichlorobenzene
GC/MS	8270C,D	1 4-Dioxane
GC/MS	8270C,D	1 4-Naphthoquinone
GC/MS	8270C,D	1 4-Phenylenediamine
GC/MS	8270C,D / SOM01.2	bis(2-Chloroisopropyl) ether (2 2`-Oxybis(1-chloropropane))
GC/MS	8270C,D / SOM01.2	2 4-Dichlorophenol
GC/MS	8270C,D / SOM01.2	2 4-Dimethylphenol
GC/MS	8270C,D / SOM01.2	2 4-Dinitrophenol

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	8270C,D / SOM01.2	2 4-Dinitrotoluene (2 4-DNT)
GC/MS	8270C,D	2 6-Dichlorophenol
GC/MS	8270C,D / SOM01.2	2 6-Dinitrotoluene (2 6-DNT)
GC/MS	8270C,D / SOM01.2	3 3`-Dichlorobenzidine
GC/MS	8270C,D	3 3'-Dimethylbenzidine
GC/MS	8270C,D / SOM01.2	2-Methyl-4 6-dinitrophenol
GC/MS	8270C,D	7,12-Dimethylphenethylamine
HPLC	8330/8330A/8330B (Analysis Only)	1 3 5-Trinitrobenzene
HPLC	8330/8330A/8330B (Analysis Only)	1 3-Dinitrobenzene
HPLC	8330/8330A/8330B (Analysis Only)	2 4 6-Trinitrotoluene
HPLC	8330/8330A/8330B (Analysis Only)	2 4-Dinitrotoluene
HPLC	8330/8330A/8330B (Analysis Only)	2 6-Dinitrotoluene
HPLC	8330/8330A/8330B (Analysis Only)	2-Amino-4 6 -dinitrotoluene
HPLC	8330/8330A/8330B (Analysis Only)	2-Nitrotoluene
HPLC	8330/8330A/8330B (Analysis Only)	3-Nitrotoluene
HPLC	8330/8330A/8330B (Analysis Only)	4-Amino-2,3-dinitrotoluene
HPLC	8330/8330A/8330B (Analysis Only)	4-Nitrotoluene
HPLC	8330/8330A/8330B (Analysis Only)	Hexahydr-1 3 5-trinitro-1 3 5-triazine (RDX)
HPLC	8330/8330A/8330B (Analysis Only)	Nitrobenzene
HPLC	8330/8330A/8330B (Analysis Only)	Nitroglycerin
HPLC	8330/8330A/8330B (Analysis Only)	Octahydro-1 3 5 7-tetrazocine (HMX)
HPLC	8330/8330A/8330B (Analysis Only)	Tetryl
CVAA	7471B/ ILM05.3	Mercury
CVAF	1631E	Low Level Mercury
ICP	6010B,C /ILM05.3	Aluminum
ICP	6010B,C /ILM05.3	Antimony
ICP	6010B,C /ILM05.3	Arsenic
ICP	6010B,C /ILM05.3	Barium
ICP	6010B,C /ILM05.3	Beryllium
ICP	6010B,C	Boron
ICP	6010B,C /ILM05.3	Cadmium

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
ICP	6010B,C /ILM05.3	Calcium
ICP	6010B,C /ILM05.3	Chromium
ICP	6010B,C /ILM05.3	Cobalt
ICP	6010B,C /ILM05.3	Copper
ICP	6010B,C /ILM05.3	Iron
ICP	6010B,C /ILM05.3	Lead
ICP	6010B,C /ILM05.3	Magnesium
ICP	6010B,C /ILM05.3	Manganese
ICP	6010B,C	Molybdenum
ICP	6010B,C /ILM05.3	Nickel
ICP	6010B,C /ILM05.3	Potassium
ICP	6010B,C /ILM05.3	Selenium
ICP	200.7	Silicon
ICP	6010B,C /ILM05.3	Silver
ICP	6010B,C /ILM05.3	Sodium
ICP	6010B,C	Strontium
ICP	6010B,C /ILM05.3	Thallium
ICP	6010B,C	Tin
ICP	6010B,C	Titanium
ICP	6010B,C /ILM05.3	Vanadium
ICP	6010B,C /ILM05.3	Zinc
ICP/MS	6020/6020A / ILM05.3	Aluminum
ICP/MS	6020/6020A / ILM05.3	Antimony
ICP/MS	6020/6020A / ILM05.3	Arsenic
ICP/MS	6020/6020A / ILM05.3	Barium
ICP/MS	6020/6020A / ILM05.3	Beryllium
ICP/MS	6020/6020A	Boron
ICP/MS	6020/6020A / ILM05.3	Cadmium
ICP/MS	6020/6020A	Calcium
ICP/MS	6020/6020A / ILM05.3	Chromium
ICP/MS	6020/6020A / ILM05.3	Cobalt
ICP/MS	6020/6020A / ILM05.3	Copper
ICP/MS	6020/6020A	Iron
ICP/MS	6020/6020A / ILM05.3	Lead
ICP/MS	6020/6020A / ILM05.3	Magnesium
ICP/MS	6020/6020A	Manganese
ICP/MS	6020/6020A	Molybdenum
ICP/MS	6020/6020A / ILM05.3	Nickel
ICP/MS	6020/6020A	Potassium
ICP/MS	6020/6020A / ILM05.3	Selenium
ICP/MS	6020/6020A / ILM05.3	Silver
ICP/MS	6020/6020A	Sodium
ICP/MS	6020/6020A	Strontium
ICP/MS	6020/6020A / ILM05.3	Thallium
ICP/MS	6020/6020A	Tin

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
ICP/MS	6020/6020A	Titanium
ICP/MS	6020/6020A / ILM05.3	Vanadium
ICP/MS	6020/6020A / ILM05.3	Zinc
IC	9056/9056A	Chloride
IC	9056/9056A	Fluoride
IC	9056/9056A	Nitrate as N
IC	9056/9056A	Nitrite as N
IC	9056/9056A	Sulfate
Gravimetric	9070A / 9071B	Oil and Grease
Physical	1010A	Ignitability
Physical	9045D	pH
Titration	Chap 7.3.4	Reactive Sulfide
TOC	Lloyd Kahn	Total organic carbon
TOC	9060A / 5310B	Total organic carbon
Turbidimetric	9038 / ASTM 516-02	Sulfate
UV/VIS	350.1 / 4500NH3 H	Ammonia as N
UV/VIS	9251 / 4500Cl E	Chloride
UV/VIS	Chap. 7.3.4	Reactive Cyanide
UV/VIS	376.3	AVS-SEM
UV/VIS	3500Fe D	Ferrous Iron
Cleanup Methods	3630C	Silica Gel
UV/VIS	7196	Chromium VI
UV/VIS	7196A	Chromium VI
UV/VIS	9012B / ILM05.3	Total cyanide
Preparation	**Method**	**Type**
Preparation	1311	Toxicity Characteristic Leaching Procedure
Preparation	1312	Synthetic Precipitation Leaching Procedure
Cleanup Methods	3660B	Sulfur
Cleanup Methods	3620C	Florsil
Cleanup Methods	3630C	Silica Gel
Cleanup Methods	3640A	GPC
Organic Preparation	3540C	Soxhlet Extraction
Organic Preparation	3545A	Pressurized Fluid Extraction
Organic Preparation	3550C	Sonication
Inorganics Preparation	3050B	Hotblock
Inorganics Preparation	3060A	Alkaline Digestion
Volatile Organics Preparation	5035/5035A	Closed System Purge and Trap

Notes:

1) This laboratory offers commercial testing service.

Approved By: _____ Date: November 4, 2009

R. Douglas Leonard
Chief Technical Officer

Issued: 11/04/09

State of Florida
Department of Health, Bureau of Laboratories

This is to certify that

E87604

KATAHDIN ANALYTICAL SERVICES, INC.
600 TECHNOLOGY WAY
SCARBOROUGH, ME 04074

has complied with Florida Administrative Code 64E-1,
for the examination of Environmental samples in the following categories

DRINKING WATER - GROUP II UNREGULATED CONTAMINANTS, DRINKING WATER - OTHER REGULATED CONTAMINANTS, DRINKING WATER - MICROBIOLOGY, DRINKING WATER - PRIMARY INORGANIC CONTAMINANTS, DRINKING WATER - SECONDARY INORGANIC CONTAMINANTS, DRINKING WATER - RADIOCHEMISTRY, DRINKING WATER - SYNTHETIC ORGANIC CONTAMINANTS, NON-POTABLE WATER - EXTRACTABLE ORGANICS, NON-POTABLE WATER - GENERAL CHEMISTRY, NON-POTABLE WATER - METALS, NON-POTABLE WATER - MICROBIOLOGY, NON-POTABLE WATER - PESTICIDES-HERBICIDES-PCB'S, NON-POTABLE WATER - VOLATILE ORGANICS, SOLID AND CHEMICAL MATERIALS - EXTRACTABLE ORGANICS, SOLID AND CHEMICAL MATERIALS - GENERAL CHEMISTRY, SOLID AND CHEMICAL MATERIALS - METALS, SOLID AND CHEMICAL MATERIALS - PESTICIDES-HERBICIDES-PCB'S, SOLID AND CHEMICAL MATERIALS - VOLATILE ORGANICS, BIOLOGICAL TISSUE - GENERAL CHEMISTRY, BIOLOGICAL TISSUE - PESTICIDES-HERBICIDES-PCB'S

Continued certification is contingent upon successful on-going compliance with the NELAC Standards and FAC Rule 64E-1 regulations. Specific methods and analytes certified are cited on the Laboratory Scope of Accreditation for this laboratory and are on file at the Bureau of Laboratories, P. O. Box 210, Jacksonville, Florida 32231. Clients and customers are urged to verify with this agency the laboratory's certification status in Florida for particular methods and analytes.

EFFECTIVE July 01, 2009 THROUGH June 30, 2010

Max Salfinger, M.D.
Chief, Bureau of Laboratories
Florida Department of Health
DH Form 1697, 7/04
NON-TRANSFERABLE E87604-13-07/01/2009
Supersedes all previously issued certificates

Laboratory Scope of Accreditation

Attachment to Certificate #: E87604-13, expiration date June 30, 2010. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: **E87604** EPA Lab Code: **ME00019** **(207) 874-2400**

E87604
Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: **Drinking Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
1,1,1-Trichloroethane	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
1,1,2,2-Tetrachloroethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
1,1,2-Trichloroethane	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
1,1-Dichloroethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
1,1-Dichloroethylene	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
1,1-Dichloropropene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
1,2,3-Trichlorobenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	4/26/2002
1,2,3-Trichloropropane	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
1,2,4-Trichlorobenzene	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
1,2,4-Trimethylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
1,2-Dibromo-3-chloropropane (DBCP)	EPA 504.1	Synthetic Organic Contaminants	NELAP	2/4/2002
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 504.1	Synthetic Organic Contaminants	NELAP	2/4/2002
1,2-Dichlorobenzene	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
1,2-Dichloroethane	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
1,2-Dichloropropane	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
1,3,5-Trimethylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
1,3-Dichlorobenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
1,3-Dichloropropane	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
1,4-Dichlorobenzene	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
2,2-Dichloropropane	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
2-Butanone (Methyl ethyl ketone, MEK)	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
2-Chlorotoluene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
2-Hexanone	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
4-Chlorotoluene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
4-Isopropyltoluene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
4-Methyl-2-pentanone (MIBK)	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Acetone	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Alkalinity as CaCO3	SM 2320 B	Primary Inorganic Contaminants	NELAP	4/26/2002
Aluminum	EPA 200.7	Secondary Inorganic Contaminants	NELAP	2/4/2002
Aluminum	EPA 200.8	Secondary Inorganic Contaminants	NELAP	4/26/2002
Amenable cyanide	SM 4500-CN G	Primary Inorganic Contaminants	NELAP	2/4/2002
Antimony	EPA 200.8	Primary Inorganic Contaminants	NELAP	2/4/2002
Arsenic	EPA 200.8	Primary Inorganic Contaminants	NELAP	4/26/2002
Barium	EPA 200.7	Primary Inorganic Contaminants	NELAP	2/4/2002
Barium	EPA 200.8	Primary Inorganic Contaminants	NELAP	2/4/2002

Laboratory Scope of Accreditation

Attachment to Certificate #: E87604-13, expiration date June 30, 2010. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: **E87604** EPA Lab Code: **ME00019** **(207) 874-2400**

E87604
Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: **Drinking Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Benzene	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
Beryllium	EPA 200.7	Primary Inorganic Contaminants	NELAP	2/4/2002
Beryllium	EPA 200.8	Primary Inorganic Contaminants	NELAP	2/4/2002
Bromobenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Bromochloromethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Bromodichloromethane	EPA 524.2	Group II Unregulated Contaminants,Other Regulated Contaminants	NELAP	2/4/2002
Bromoform	EPA 524.2	Group II Unregulated Contaminants,Other Regulated Contaminants	NELAP	2/4/2002
Cadmium	EPA 200.7	Primary Inorganic Contaminants	NELAP	2/4/2002
Cadmium	EPA 200.8	Primary Inorganic Contaminants	NELAP	2/4/2002
Calcium	CA-628-01(EPA 200.8)/ICP-MS	Primary Inorganic Contaminants	NELAP	11/7/2006
Calcium	EPA 200.7	Primary Inorganic Contaminants	NELAP	2/4/2002
Carbon disulfide	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Carbon tetrachloride	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
Chloride	EPA 300.0	Secondary Inorganic Contaminants	NELAP	4/26/2002
Chloride	EPA 325.2	Secondary Inorganic Contaminants	NELAP	2/4/2002
Chlorobenzene	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
Chloroethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Chloroform	EPA 524.2	Group II Unregulated Contaminants,Other Regulated Contaminants	NELAP	2/4/2002
Chromium	EPA 200.7	Primary Inorganic Contaminants	NELAP	2/4/2002
Chromium	EPA 200.8	Primary Inorganic Contaminants	NELAP	2/4/2002
cis-1,2-Dichloroethylene	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
cis-1,3-Dichloropropene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Color	EPA 110.2	Secondary Inorganic Contaminants	NELAP	2/4/2002
Color	SM 2120 B	Secondary Inorganic Contaminants	NELAP	4/17/2007
Copper	EPA 200.7	Secondary Inorganic Contaminants,Primary Inorganic Contaminants	NELAP	2/4/2002
Copper	EPA 200.8	Secondary Inorganic Contaminants,Primary Inorganic Contaminants	NELAP	2/4/2002
Cyanide	EPA 335.4	Primary Inorganic Contaminants	NELAP	2/4/2002
Dibromochloromethane	EPA 524.2	Group II Unregulated Contaminants,Other Regulated Contaminants	NELAP	2/4/2002
Dibromomethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002

Laboratory Scope of Accreditation

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State Laboratory ID: **E87604** EPA Lab Code: **ME00019** **(207) 874-2400**

E87604
Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: **Drinking Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Dichlorodifluoromethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Dichloromethane (DCM, Methylene chloride)	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
Diethyl ether	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Escherichia coli	COLITAG	Microbiology	NELAP	11/7/2006
Ethylbenzene	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
Fluoride	SM 4500 F-C	Secondary Inorganic Contaminants,Primary Inorganic Contaminants	NELAP	2/4/2002
Heterotrophic plate count	SIMPLATE	Microbiology	NELAP	11/7/2006
Hexachlorobutadiene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Iron	CA-628-01(EPA 200.8)/ICP-MS	Primary Inorganic Contaminants	NELAP	11/7/2006
Iron	EPA 200.7	Secondary Inorganic Contaminants	NELAP	2/4/2002
Isopropylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Lead	EPA 200.8	Primary Inorganic Contaminants	NELAP	2/4/2002
Magnesium	CA-628-01(EPA 200.8)/ICP-MS	Primary Inorganic Contaminants	NELAP	11/7/2006
Magnesium	EPA 200.7	Primary Inorganic Contaminants	NELAP	4/26/2002
Manganese	EPA 200.7	Secondary Inorganic Contaminants	NELAP	2/4/2002
Manganese	EPA 200.8	Secondary Inorganic Contaminants	NELAP	2/4/2002
Mercury	EPA 245.1	Primary Inorganic Contaminants	NELAP	2/4/2002
Methyl bromide (Bromomethane)	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Methyl chloride (Chloromethane)	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Methyl tert-butyl ether (MTBE)	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Naphthalene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
n-Butylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Nickel	EPA 200.7	Primary Inorganic Contaminants	NELAP	2/4/2002
Nickel	EPA 200.8	Primary Inorganic Contaminants	NELAP	2/4/2002
Nitrate	EPA 300.0	Primary Inorganic Contaminants	NELAP	4/26/2002
Nitrate	EPA 353.2	Primary Inorganic Contaminants	NELAP	2/4/2002
Nitrate-nitrite	EPA 300.0	Primary Inorganic Contaminants	NELAP	4/26/2002
Nitrite	EPA 300.0	Primary Inorganic Contaminants	NELAP	4/26/2002
Nitrite	EPA 353.2	Primary Inorganic Contaminants	NELAP	2/4/2002
n-Propylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Orthophosphate as P	EPA 300.0	Primary Inorganic Contaminants	NELAP	7/30/2004
Perchlorate	EPA 314.0	Secondary Inorganic Contaminants	NELAP	7/30/2004
pH	EPA 150.1	Primary Inorganic Contaminants,Secondary Inorganic Contaminants	NELAP	2/4/2002

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Matrix: **Drinking Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
pH	SM 4500-H+-B	Secondary Inorganic Contaminants	NELAP	4/17/2007
sec-Butylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Selenium	EPA 200.8	Primary Inorganic Contaminants	NELAP	2/4/2002
Silica as SiO2	EPA 200.7	Primary Inorganic Contaminants	NELAP	2/4/2002
Silver	EPA 200.7	Secondary Inorganic Contaminants	NELAP	2/4/2002
Silver	EPA 200.8	Secondary Inorganic Contaminants	NELAP	2/4/2002
Sodium	CA-628-01(EPA 200.8)/ICP-MS	Primary Inorganic Contaminants	NELAP	11/7/2006
Sodium	EPA 200.7	Primary Inorganic Contaminants	NELAP	4/26/2002
Styrene	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
Sulfate	ASTM D516-02	Secondary Inorganic Contaminants	NELAP	5/8/2009
Sulfate	ASTM D516-90	Secondary Inorganic Contaminants	NELAP	5/8/2009
Sulfate	EPA 300.0	Secondary Inorganic Contaminants,Primary Inorganic Contaminants	NELAP	2/4/2002
tert-Butylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Tetrachloroethylene (Perchloroethylene)	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
Tetrahydrofuran (THF)	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Thallium	EPA 200.8	Primary Inorganic Contaminants	NELAP	2/4/2002
Toluene	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
Total coliforms	COLITAG	Microbiology	NELAP	11/7/2006
Total dissolved solids	EPA 160.1	Secondary Inorganic Contaminants	NELAP	2/4/2002
Total dissolved solids	SM 2540 C	Secondary Inorganic Contaminants	NELAP	2/4/2002
Total nitrate-nitrite	EPA 353.2	Primary Inorganic Contaminants	NELAP	2/4/2002
Total organic carbon	SM 5310B	Primary Inorganic Contaminants	NELAP	5/8/2009
Total trihalomethanes	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
trans-1,2-Dichloroethylene	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
trans-1,3-Dichloropropylene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Trichloroethene (Trichloroethylene)	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
Trichlorofluoromethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Turbidity	EPA 180.1	Secondary Inorganic Contaminants	NELAP	2/4/2002
Uranium	EPA 200.8	Radiochemistry	NELAP	11/7/2006
Vinyl chloride	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
Xylene (total)	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
Zinc	EPA 200.7	Secondary Inorganic Contaminants	NELAP	2/4/2002
Zinc	EPA 200.8	Secondary Inorganic Contaminants	NELAP	2/4/2002

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State Laboratory ID: **E87604** EPA Lab Code: **ME00019** **(207) 874-2400**

E87604
Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1,1-Trichloroethane	EPA 624	Volatile Organics	NELAP	2/4/2002
1,1,1-Trichloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1,1-Trichloroethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,1,2,2-Tetrachloroethane	EPA 624	Volatile Organics	NELAP	2/4/2002
1,1,2,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1,2,2-Tetrachloroethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,1,2,2-Tetrachloroethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1,2,2-Tetrachloroethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1,2-Trichloro-1,2,2-trifluoroethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,1,2-Trichloro-1,2,2-trifluoroethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1,2-Trichloro-1,2,2-trifluoroethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1,2-Trichloroethane	EPA 624	Volatile Organics	NELAP	2/4/2002
1,1,2-Trichloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1,2-Trichloroethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,1,2-Trichloroethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1,2-Trichloroethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1-Dichloroethane	EPA 624	Volatile Organics	NELAP	2/4/2002
1,1-Dichloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1-Dichloroethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,1-Dichloroethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1-Dichloroethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1-Dichloroethylene	EPA 624	Volatile Organics	NELAP	2/4/2002
1,1-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1-Dichloroethylene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,1-Dichloroethylene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1-Dichloroethylene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1-Dichloropropene	EPA 8260	Volatile Organics	NELAP	7/1/2003

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Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,2,3-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2,3-Trichlorobenzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2,3-Trichlorobenzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2,3-Trichloropropane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2,4,5-Tetrachlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,2,4,5-Tetrachlorobenzene	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
1,2,4-Trichlorobenzene	EPA 625	Extractable Organics	NELAP	2/4/2002
1,2,4-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2,4-Trichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,2,4-Trichlorobenzene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,2,4-Trichlorobenzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2,4-Trichlorobenzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2,4-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dibromo-3-chloropropane (DBCP)	EPA 504	Volatile Organics	NELAP	2/4/2002
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8011	Volatile Organics	NELAP	7/1/2003
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dibromo-3-chloropropane (DBCP)	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,2-Dibromo-3-chloropropane (DBCP)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dibromo-3-chloropropane (DBCP) (with SIM)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 504	Volatile Organics	NELAP	2/4/2002
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8011	Volatile Organics	NELAP	7/1/2003
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dibromoethane (EDB, Ethylene dibromide)	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,2-Dibromoethane (EDB, Ethylene dibromide)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dibromoethane (EDB, Ethylene dibromide) (with SIM)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	2/4/2002
1,2-Dichlorobenzene	EPA 625	Extractable Organics	NELAP	2/4/2002
1,2-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003

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E87604
Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,2-Dichlorobenzene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,2-Dichlorobenzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dichlorobenzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dichloroethane	EPA 624	Volatile Organics	NELAP	2/4/2002
1,2-Dichloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dichloroethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,2-Dichloroethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dichloroethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dichloropropane	EPA 624	Volatile Organics	NELAP	2/4/2002
1,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dichloropropane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,2-Dichloropropane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dichloropropane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Diphenylhydrazine	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,3,5-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,3,5-Trinitrobenzene (1,3,5-TNB)	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,3,5-Trinitrobenzene (1,3,5-TNB)	EPA 8330	Extractable Organics	NELAP	7/30/2004
1,3-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	2/4/2002
1,3-Dichlorobenzene	EPA 625	Extractable Organics	NELAP	2/4/2002
1,3-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,3-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,3-Dichlorobenzene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,3-Dichlorobenzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,3-Dichlorobenzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,3-Dichloropropane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,3-Dinitrobenzene (1,3-DNB)	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,3-Dinitrobenzene (1,3-DNB)	EPA 8330	Extractable Organics	NELAP	7/30/2004
1,4-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	2/4/2002
1,4-Dichlorobenzene	EPA 625	Extractable Organics	NELAP	2/4/2002
1,4-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003

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Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,4-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,4-Dichlorobenzene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,4-Dichlorobenzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,4-Dichlorobenzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,4-Dioxane (1,4-Diethyleneoxide)	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,4-Dioxane (1,4-Diethyleneoxide)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,4-Dioxane (1,4-Diethyleneoxide) (without SIM)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,4-Naphthoquinone	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,4-Phenylenediamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
1-Naphthylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (BZ 206)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',3,3',4,4',5,6-Octachlorobiphenyl (BZ 195)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ 170)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',3,3',4,4'-Hexachlorobiphenyl (BZ 128)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,2',3,4,4',5,5'-Heptachlorobiphenyl (BZ 180)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',3,4,4',5',6-Heptachlorobiphenyl (BZ 183)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',3,4,4',5'-Hexachlorobiphenyl (BZ 138)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',3,4,4',6,6'-Heptachlorobiphenyl (BZ 184)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,2',3,4',5,5',6-Heptachlorobiphenyl (BZ 187)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',3,4,5'-Pentachlorobiphenyl (BZ 87)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',3,5'-Tetrachlorobiphenyl (BZ 44)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',4,4',5,5'-Hexachlorobiphenyl (BZ 153)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',4,5,5'-Pentachlorobiphenyl (BZ 101)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',4,5'-Tetrachlorobiphenyl (BZ 49)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,2',5,5'-Tetrachlorobiphenyl (BZ 52)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',5-Trichlorobiphenyl (BZ 18)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	7/1/2003
2,3,3',4,4',5,5'-Heptachlorobiphenyl (BZ 189)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,3,3',4,4',5-Hexachlorobiphenyl (BZ 156)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,3,3',4,4',5'-Hexachlorobiphenyl (BZ 157)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,3,3',4,4'-Pentachlorobiphenyl (BZ 105)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,3',4,4',5,5'-Hexachlorobiphenyl (BZ 167)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,3,4,4',5-Pentachlorobiphenyl (BZ 114)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009

Laboratory Scope of Accreditation

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State Laboratory ID: **E87604** EPA Lab Code: **ME00019** **(207) 874-2400**

E87604
Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,3',4,4',5-Pentachlorobiphenyl (BZ 118)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,3',4,4',5'-Pentachlorobiphenyl (BZ 123)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,3',4,4'-Tetrachlorobiphenyl (BZ 66)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,3,4,6-Tetrachlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,3,4,6-Tetrachlorophenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2,4,4'-Trichlorobiphenyl (BZ 28)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,4,5-T	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,4,5-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4,5-Trichlorophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2,4,5-Trichlorophenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2,4,6-Trichlorophenol	EPA 625	Extractable Organics	NELAP	2/4/2002
2,4,6-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4,6-Trichlorophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2,4,6-Trichlorophenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2,4,6-Trinitrotoluene (2,4,6-TNT)	EPA 8330	Extractable Organics	NELAP	7/30/2004
2,4-D	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,4-DB	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,4'-Dichlorobiphenyl (BZ 8)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,4-Dichlorophenol	EPA 625	Extractable Organics	NELAP	2/4/2002
2,4-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4-Dichlorophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2,4-Dichlorophenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2,4-Dimethylphenol	EPA 625	Extractable Organics	NELAP	2/4/2002
2,4-Dimethylphenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4-Dimethylphenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2,4-Dimethylphenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2,4-Dinitrophenol	EPA 625	Extractable Organics	NELAP	2/4/2002
2,4-Dinitrophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4-Dinitrophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2,4-Dinitrophenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2,4-Dinitrotoluene (2,4-DNT)	EPA 625	Extractable Organics	NELAP	2/4/2002
2,4-Dinitrotoluene (2,4-DNT)	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4-Dinitrotoluene (2,4-DNT)	EPA 8330	Extractable Organics	NELAP	7/30/2004
2,4-Dinitrotoluene (2,4-DNT)	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004

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E87604
Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,4-Dinitrotoluene (2,4-DNT)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2,6-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,6-Dinitrotoluene (2,6-DNT)	EPA 625	Extractable Organics	NELAP	2/4/2002
2,6-Dinitrotoluene (2,6-DNT)	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,6-Dinitrotoluene (2,6-DNT)	EPA 8330	Extractable Organics	NELAP	7/30/2004
2,6-Dinitrotoluene (2,6-DNT)	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2-Acetylaminofluorene	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Amino-4,6-dinitrotoluene (2-am-dnt)	EPA 8330	Extractable Organics	NELAP	7/30/2004
2-Butanone (Methyl ethyl ketone, MEK)	EPA 8260	Volatile Organics	NELAP	7/1/2003
2-Butanone (Methyl ethyl ketone, MEK)	OLM04.3-Exhibit D	Volatile Organics	NELAP	5/12/2005
2-Butanone (Methyl ethyl ketone, MEK)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
2-Butanone (Methyl ethyl ketone, MEK)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
2-Chloroethyl vinyl ether	EPA 624	Volatile Organics	NELAP	2/4/2002
2-Chloroethyl vinyl ether	EPA 8260	Volatile Organics	NELAP	7/1/2003
2-Chloronaphthalene	EPA 625	Extractable Organics	NELAP	2/4/2002
2-Chloronaphthalene	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Chloronaphthalene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2-Chloronaphthalene	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2-Chlorophenol	EPA 625	Extractable Organics	NELAP	2/4/2002
2-Chlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Chlorophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2-Chlorophenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	7/1/2003
2-Hexanone	EPA 8260	Volatile Organics	NELAP	7/1/2003
2-Hexanone	OLM04.3-Exhibit D	Volatile Organics	NELAP	5/12/2005
2-Hexanone	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
2-Hexanone	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
2-Methyl-4,6-dinitrophenol	EPA 625	Extractable Organics	NELAP	2/4/2002
2-Methyl-4,6-dinitrophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Methyl-4,6-dinitrophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2-Methylnaphthalene	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Methylnaphthalene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004

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E87604
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Scarborough, ME 04074

Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
2-Methylnaphthalene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2-Methylphenol (o-Cresol)	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Methylphenol (o-Cresol)	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2-Methylphenol (o-Cresol)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2-Naphthylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Nitroaniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Nitroaniline	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2-Nitrophenol	EPA 625	Extractable Organics	NELAP	2/4/2002
2-Nitrophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Nitrophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2-Nitrophenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	7/30/2004
2-Picoline (2-Methylpyridine)	EPA 8270	Extractable Organics	NELAP	7/1/2003
3,3',4,4',5,5'-Hexachlorobiphenyl (BZ 169)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
3,3',4,4',5-Pentachlorobiphenyl (BZ 126)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
3,3',4,4'-Tetrachlorobiphenyl (BZ 77)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
3,3'-Dichlorobenzidine	EPA 625	Extractable Organics	NELAP	2/4/2002
3,3'-Dichlorobenzidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
3,3'-Dichlorobenzidine	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
3,3'-Dichlorobenzidine	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
3,3'-Dimethylbenzidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
3,4,4',5-Tetrachlorobiphenyl (BZ 81)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
3-Methylcholanthrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
3-Nitroaniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
3-Nitroaniline	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
3-Nitroaniline	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
3-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	7/30/2004
4,4'-DDD	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
4,4'-DDD	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
4,4'-DDD	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
4,4'-DDD	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
4,4'-DDE	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
4,4'-DDE	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
4,4'-DDE	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004

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600 Technology Way
Scarborough, ME 04074

Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
4,4'-DDE	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
4,4'-DDT	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
4,4'-DDT	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
4,4'-DDT	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
4,4'-DDT	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
4-Amino-2,6-dinitrotoluene (4-am-dnt)	EPA 8330	Extractable Organics	NELAP	7/30/2004
4-Aminobiphenyl	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Bromophenyl phenyl ether	EPA 625	Extractable Organics	NELAP	2/4/2002
4-Bromophenyl phenyl ether	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Bromophenyl phenyl ether	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
4-Bromophenyl phenyl ether	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
4-Chloro-3-methylphenol	EPA 625	Extractable Organics	NELAP	2/4/2002
4-Chloro-3-methylphenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Chloro-3-methylphenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
4-Chloro-3-methylphenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
4-Chloroaniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Chloroaniline	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
4-Chloroaniline	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
4-Chlorophenyl phenylether	EPA 625	Extractable Organics	NELAP	2/4/2002
4-Chlorophenyl phenylether	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Chlorophenyl phenylether	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
4-Chlorophenyl phenylether	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
4-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	7/1/2003
4-Dimethyl aminoazobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Methyl-2-pentanone (MIBK)	EPA 8260	Volatile Organics	NELAP	7/1/2003
4-Methyl-2-pentanone (MIBK)	OLM04.3-Exhibit D	Volatile Organics	NELAP	5/12/2005
4-Methyl-2-pentanone (MIBK)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
4-Methyl-2-pentanone (MIBK)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
4-Methylphenol (p-Cresol)	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Methylphenol (p-Cresol)	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
4-Methylphenol (p-Cresol)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009

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Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
4-Nitroaniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Nitroaniline	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
4-Nitroaniline	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
4-Nitrophenol	EPA 625	Extractable Organics	NELAP	2/4/2002
4-Nitrophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Nitrophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
4-Nitrophenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
4-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	7/30/2004
5-Nitro-o-toluidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
7,12-Dimethylbenz(a) anthracene	EPA 8270	Extractable Organics	NELAP	7/1/2003
a-a-Dimethylphenethylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
Acenaphthene	EPA 625	Extractable Organics	NELAP	2/4/2002
Acenaphthene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Acenaphthene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Acenaphthene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Acenaphthylene	EPA 625	Extractable Organics	NELAP	2/4/2002
Acenaphthylene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Acenaphthylene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Acenaphthylene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Acetone	EPA 8260	Volatile Organics	NELAP	7/1/2003
Acetone	OLM04.3-Exhibit D	Volatile Organics	NELAP	5/12/2005
Acetone	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Acetone	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Acetonitrile	EPA 8260	Volatile Organics	NELAP	7/1/2003
Acetophenone	EPA 8270	Extractable Organics	NELAP	7/1/2003
Acetophenone	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Acetophenone	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Acidity, as CaCO3	EPA 305.1	General Chemistry	NELAP	2/4/2002
Acidity, as CaCO3	SM 2310 B (4A)	General Chemistry	NELAP	4/17/2007
Acrolein (Propenal)	EPA 624	Volatile Organics	NELAP	4/26/2002
Acrolein (Propenal)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Acrylonitrile	EPA 624	Volatile Organics	NELAP	4/26/2002

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Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Acrylonitrile	EPA 8260	Volatile Organics	NELAP	7/1/2003
Aldrin	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Aldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aldrin	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aldrin	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Alkalinity as CaCO3	EPA 310.1	General Chemistry	NELAP	2/4/2002
Alkalinity as CaCO3	EPA 310.2	General Chemistry	NELAP	7/30/2004
Alkalinity as CaCO3	SM 2320 B	General Chemistry	NELAP	2/4/2002
Allyl chloride (3-Chloropropene)	EPA 8260	Volatile Organics	NELAP	7/1/2003
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
alpha-BHC (alpha-Hexachlorocyclohexane)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
alpha-BHC (alpha-Hexachlorocyclohexane)	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
alpha-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
alpha-Chlordane	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
alpha-Chlordane	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aluminum	EPA 200.7	Metals	NELAP	2/4/2002
Aluminum	EPA 200.8	Metals	NELAP	2/4/2002
Aluminum	EPA 6010	Metals	NELAP	7/1/2003
Aluminum	EPA 6020	Metals	NELAP	7/1/2003
Aluminum	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Aluminum	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006
Amenable cyanide	EPA 335.1	General Chemistry	NELAP	2/4/2002
Amenable cyanide	EPA 335.4	General Chemistry	NELAP	2/4/2002
Amenable cyanide	EPA 9012	General Chemistry	NELAP	7/1/2003
Amenable cyanide	SM 4500-CN G	General Chemistry	NELAP	2/4/2002
Ammonia as N	EPA 350.1	General Chemistry	NELAP	2/4/2002
Ammonia as N	SM 4500-NH3 H	General Chemistry	NELAP	2/4/2002
Aniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
Anthracene	EPA 625	Extractable Organics	NELAP	2/4/2002
Anthracene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Anthracene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Anthracene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Antimony	EPA 200.7	Metals	NELAP	2/4/2002

Laboratory Scope of Accreditation

Attachment to Certificate #: E87604-13, expiration date June 30, 2010. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: **E87604** EPA Lab Code: **ME00019** **(207) 874-2400**

E87604
Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Antimony	EPA 200.8	Metals	NELAP	2/4/2002
Antimony	EPA 6010	Metals	NELAP	7/1/2003
Antimony	EPA 6020	Metals	NELAP	7/1/2003
Antimony	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Antimony	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006
Aramite	EPA 8270	Extractable Organics	NELAP	7/1/2003
Aroclor-1016 (PCB-1016)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Aroclor-1016 (PCB-1016)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1016 (PCB-1016)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1016 (PCB-1016)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aroclor-1221 (PCB-1221)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Aroclor-1221 (PCB-1221)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1221 (PCB-1221)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1221 (PCB-1221)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aroclor-1232 (PCB-1232)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Aroclor-1232 (PCB-1232)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1232 (PCB-1232)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1232 (PCB-1232)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aroclor-1242 (PCB-1242)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Aroclor-1242 (PCB-1242)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1242 (PCB-1242)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1242 (PCB-1242)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aroclor-1248 (PCB-1248)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Aroclor-1248 (PCB-1248)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1248 (PCB-1248)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1248 (PCB-1248)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aroclor-1254 (PCB-1254)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Aroclor-1254 (PCB-1254)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1254 (PCB-1254)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1254 (PCB-1254)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aroclor-1260 (PCB-1260)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Aroclor-1260 (PCB-1260)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1260 (PCB-1260)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004

Laboratory Scope of Accreditation

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State Laboratory ID: **E87604** EPA Lab Code: **ME00019** **(207) 874-2400**

E87604
Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Aroclor-1260 (PCB-1260)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aroclor-1262 (PCB-1262)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aroclor-1268 (PCB-1268)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Arsenic	EPA 200.7	Metals	NELAP	2/4/2002
Arsenic	EPA 200.8	Metals	NELAP	2/4/2002
Arsenic	EPA 6010	Metals	NELAP	7/1/2003
Arsenic	EPA 6020	Metals	NELAP	2/4/2002
Arsenic	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Arsenic	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006
Atrazine	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Atrazine	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Barium	EPA 200.7	Metals	NELAP	2/4/2002
Barium	EPA 200.8	Metals	NELAP	2/4/2002
Barium	EPA 6010	Metals	NELAP	7/1/2003
Barium	EPA 6020	Metals	NELAP	7/1/2003
Barium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Barium	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006
Benzaldehyde	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Benzaldehyde	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Benzene	EPA 624	Volatile Organics	NELAP	2/4/2002
Benzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Benzene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Benzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Benzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Benzidine	EPA 625	Extractable Organics	NELAP	2/4/2002
Benzidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(a)anthracene	EPA 625	Extractable Organics	NELAP	2/4/2002
Benzo(a)anthracene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(a)anthracene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Benzo(a)anthracene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Benzo(a)pyrene	EPA 625	Extractable Organics	NELAP	2/4/2002

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Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Benzo(a)pyrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(a)pyrene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Benzo(a)pyrene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Benzo(b)fluoranthene	EPA 625	Extractable Organics	NELAP	2/4/2002
Benzo(b)fluoranthene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(b)fluoranthene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Benzo(b)fluoranthene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Benzo(g,h,i)perylene	EPA 625	Extractable Organics	NELAP	2/4/2002
Benzo(g,h,i)perylene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(g,h,i)perylene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Benzo(g,h,i)perylene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Benzo(k)fluoranthene	EPA 625	Extractable Organics	NELAP	2/4/2002
Benzo(k)fluoranthene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(k)fluoranthene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Benzo(k)fluoranthene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Benzoic acid	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzyl alcohol	EPA 8270	Extractable Organics	NELAP	7/1/2003
Beryllium	EPA 200.7	Metals	NELAP	2/4/2002
Beryllium	EPA 200.8	Metals	NELAP	2/4/2002
Beryllium	EPA 6010	Metals	NELAP	7/1/2003
Beryllium	EPA 6020	Metals	NELAP	7/1/2003
Beryllium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Beryllium	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006
beta-BHC (beta-Hexachlorocyclohexane)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
beta-BHC (beta-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
beta-BHC (beta-Hexachlorocyclohexane)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
beta-BHC (beta-Hexachlorocyclohexane)	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Biochemical oxygen demand	EPA 405.1	General Chemistry	NELAP	2/4/2002
Biochemical oxygen demand	SM 5210 B	General Chemistry	NELAP	2/4/2002
Biphenyl	EPA 8270	Extractable Organics	NELAP	5/8/2009
Biphenyl	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Biphenyl	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
bis(2-Chloroethoxy)methane	EPA 625	Extractable Organics	NELAP	2/4/2002

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Katahdin Analytical Services, Inc.
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Scarborough, ME 04074

Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
bis(2-Chloroethoxy)methane	EPA 8270	Extractable Organics	NELAP	7/1/2003
bis(2-Chloroethoxy)methane	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
bis(2-Chloroethoxy)methane	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
bis(2-Chloroethyl) ether	EPA 625	Extractable Organics	NELAP	2/4/2002
bis(2-Chloroethyl) ether	EPA 8270	Extractable Organics	NELAP	7/1/2003
bis(2-Chloroethyl) ether	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
bis(2-Chloroethyl) ether	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 625	Extractable Organics	NELAP	2/4/2002
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 8270	Extractable Organics	NELAP	7/1/2003
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 625	Extractable Organics	NELAP	2/4/2002
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 8270	Extractable Organics	NELAP	7/1/2003
bis(2-Ethylhexyl) phthalate (DEHP)	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
bis(2-Ethylhexyl) phthalate (DEHP)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Boron	CA-627-02(EPA6020)/ICP-MS	Metals	NELAP	11/7/2006
Boron	CA-628-01(EPA 200.8)/ICP-MS	Metals	NELAP	11/7/2006
Boron	EPA 200.7	Metals	NELAP	2/4/2002
Boron	EPA 6010	Metals	NELAP	7/1/2003
Bromide	EPA 300.0	General Chemistry	NELAP	2/4/2002
Bromide	EPA 9056	General Chemistry	NELAP	7/1/2003
Bromobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Bromochloromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Bromochloromethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Bromochloromethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Bromodichloromethane	EPA 624	Volatile Organics	NELAP	2/4/2002
Bromodichloromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Bromodichloromethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Bromodichloromethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009

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Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Bromodichloromethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Bromoform	EPA 624	Volatile Organics	NELAP	2/4/2002
Bromoform	EPA 8260	Volatile Organics	NELAP	7/1/2003
Bromoform	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Bromoform	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Bromoform	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Butyl benzyl phthalate	EPA 625	Extractable Organics	NELAP	2/4/2002
Butyl benzyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Butyl benzyl phthalate	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Butyl benzyl phthalate	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Cadmium	EPA 200.7	Metals	NELAP	2/4/2002
Cadmium	EPA 200.8	Metals	NELAP	2/4/2002
Cadmium	EPA 6010	Metals	NELAP	7/1/2003
Cadmium	EPA 6020	Metals	NELAP	2/4/2002
Cadmium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Cadmium	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006
Calcium	EPA 200.7	Metals	NELAP	2/4/2002
Calcium	EPA 6010	Metals	NELAP	7/1/2003
Calcium	EPA 6020	Metals	NELAP	11/7/2006
Calcium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Caprolactam	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Caprolactam	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Carbazole	EPA 8270	Extractable Organics	NELAP	7/1/2003
Carbazole	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Carbazole	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Carbon disulfide	EPA 8260	Volatile Organics	NELAP	7/1/2003
Carbon disulfide	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Carbon disulfide	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Carbon disulfide	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Carbon tetrachloride	EPA 624	Volatile Organics	NELAP	2/4/2002

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Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Carbon tetrachloride	EPA 8260	Volatile Organics	NELAP	7/1/2003
Carbon tetrachloride	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Carbon tetrachloride	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Carbon tetrachloride	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Carbonaceous BOD (CBOD)	SM 5210 B	General Chemistry	NELAP	4/26/2002
Chemical oxygen demand	EPA 410.4	General Chemistry	NELAP	2/4/2002
Chemical oxygen demand	HACH 8000	General Chemistry	NELAP	4/26/2002
Chlordane (tech.)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Chlordane (tech.)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Chloride	EPA 300.0	General Chemistry	NELAP	2/4/2002
Chloride	EPA 325.2	General Chemistry	NELAP	2/4/2002
Chloride	EPA 9056	General Chemistry	NELAP	7/1/2003
Chloride	EPA 9251	General Chemistry	NELAP	7/1/2003
Chloride	SM 4500 Cl- E	General Chemistry	NELAP	2/4/2002
Chlorobenzene	EPA 624	Volatile Organics	NELAP	2/4/2002
Chlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Chlorobenzene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Chlorobenzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Chlorobenzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Chlorobenzilate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Chloroethane	EPA 624	Volatile Organics	NELAP	2/4/2002
Chloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Chloroethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Chloroethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Chloroethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Chloroform	EPA 624	Volatile Organics	NELAP	2/4/2002
Chloroform	EPA 8260	Volatile Organics	NELAP	7/1/2003
Chloroform	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Chloroprene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Chromium	EPA 200.7	Metals	NELAP	2/4/2002
Chromium	EPA 200.8	Metals	NELAP	4/26/2002

Laboratory Scope of Accreditation

Attachment to Certificate #: E87604-13, expiration date June 30, 2010. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: **E87604** EPA Lab Code: **ME00019** **(207) 874-2400**

E87604
Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Chromium	EPA 6010	Metals	NELAP	7/1/2003
Chromium	EPA 6020	Metals	NELAP	4/26/2002
Chromium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Chromium	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006
Chromium VI	EPA 7196	General Chemistry	NELAP	7/1/2003
Chromium VI	SM 3500-Cr D (18th/19th Ed.)/UV-VIS	General Chemistry	NELAP	4/26/2002
Chrysene	EPA 625	Extractable Organics	NELAP	2/4/2002
Chrysene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Chrysene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Chrysene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
cis-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	7/1/2003
cis-1,2-Dichloroethylene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
cis-1,2-Dichloroethylene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
cis-1,2-Dichloroethylene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
cis-1,3-Dichloropropene	EPA 624	Volatile Organics	NELAP	2/4/2002
cis-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	7/1/2003
cis-1,3-Dichloropropene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
cis-1,3-Dichloropropene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
cis-1,3-Dichloropropene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Cobalt	EPA 200.7	Metals	NELAP	2/4/2002
Cobalt	EPA 200.8	Metals	NELAP	2/4/2002
Cobalt	EPA 6010	Metals	NELAP	7/1/2003
Cobalt	EPA 6020	Metals	NELAP	7/1/2003
Cobalt	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Cobalt	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006
Color	EPA 110.2	General Chemistry	NELAP	4/26/2002
Color	SM 2120 B	General Chemistry	NELAP	2/4/2002
Conductivity	EPA 120.1	General Chemistry	NELAP	2/4/2002
Conductivity	SM 2510 B	General Chemistry	NELAP	2/4/2002
Copper	EPA 200.7	Metals	NELAP	2/4/2002
Copper	EPA 200.8	Metals	NELAP	2/4/2002

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Katahdin Analytical Services, Inc.
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Scarborough, ME 04074

Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Copper	EPA 6010	Metals	NELAP	7/1/2003
Copper	EPA 6020	Metals	NELAP	2/4/2002
Copper	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Copper	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006
Cyanide	EPA 335.3	General Chemistry	NELAP	2/4/2002
Cyanide	EPA 335.4	General Chemistry	NELAP	2/4/2002
Cyclohexane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Cyclohexane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Cyclohexane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Dalapon	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Decachlorobiphenyl (BZ 209)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
delta-BHC	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
delta-BHC	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
delta-BHC	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
delta-BHC	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Diallate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dibenz(a,h)anthracene	EPA 625	Extractable Organics	NELAP	2/4/2002
Dibenz(a,h)anthracene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Dibenz(a,h)anthracene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Dibenz(a,h)anthracene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Dibenzofuran	EPA 8270	Extractable Organics	NELAP	7/1/2003
Dibenzofuran	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Dibenzofuran	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Dibromochloromethane	EPA 624	Volatile Organics	NELAP	2/4/2002
Dibromochloromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Dibromochloromethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Dibromochloromethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Dibromochloromethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Dibromomethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Dicamba	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dichlorodifluoromethane	EPA 624	Volatile Organics	NELAP	2/4/2002

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Dichlorodifluoromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Dichlorodifluoromethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Dichlorodifluoromethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Dichlorodifluoromethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Dichloroprop (Dichlorprop)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dieldrin	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Dieldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dieldrin	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Dieldrin	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Diesel range organics (DRO)	EPA 8015	Extractable Organics	NELAP	7/1/2003
Diesel range organics (DRO)	MA-EPH	Extractable Organics	NELAP	7/1/2003
Diesel range organics (DRO)	MEDEP 4.1.25	Extractable Organics	NELAP	7/1/2003
Diethyl ether	EPA 8260	Volatile Organics	NELAP	7/1/2003
Diethyl phthalate	EPA 625	Extractable Organics	NELAP	2/4/2002
Diethyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Diethyl phthalate	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Diethyl phthalate	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Di-isopropylether (DIPE)	EPA 8260	Volatile Organics	NELAP	5/8/2009
Dimethoate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dimethyl phthalate	EPA 625	Extractable Organics	NELAP	2/4/2002
Dimethyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Dimethyl phthalate	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Di-n-butyl phthalate	EPA 625	Extractable Organics	NELAP	2/4/2002
Di-n-butyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Di-n-butyl phthalate	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Di-n-butyl phthalate	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Di-n-octyl phthalate	EPA 625	Extractable Organics	NELAP	2/4/2002
Di-n-octyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Di-n-octyl phthalate	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Di-n-octyl phthalate	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endosulfan I	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Endosulfan I	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003

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Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Endosulfan I	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endosulfan I	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Endosulfan II	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Endosulfan II	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endosulfan II	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endosulfan II	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Endosulfan sulfate	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Endosulfan sulfate	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endosulfan sulfate	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endosulfan sulfate	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Endrin	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Endrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endrin	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endrin	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Endrin aldehyde	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Endrin aldehyde	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endrin aldehyde	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endrin aldehyde	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Endrin ketone	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endrin ketone	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endrin ketone	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Escherichia coli	SM 9223 B /QUANTI-TRAY	Microbiology	NELAP	9/4/2007
Ethanol	EPA 8015	Volatile Organics	NELAP	7/1/2003
Ethyl methacrylate	EPA 8260	Volatile Organics	NELAP	7/1/2003
Ethyl methanesulfonate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Ethylbenzene	EPA 624	Volatile Organics	NELAP	2/4/2002
Ethylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Ethylbenzene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Ethylbenzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Ethylbenzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Ethylene glycol	EPA 8015	Volatile Organics	NELAP	5/12/2005

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Ethyl-t-butylether (ETBE)	EPA 8260	Volatile Organics	NELAP	5/8/2009
Famphur	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Fecal coliforms	SM 9222 D	Microbiology	NELAP	7/30/2004
Fluoranthene	EPA 625	Extractable Organics	NELAP	2/4/2002
Fluoranthene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Fluoranthene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Fluoranthene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Fluorene	EPA 625	Extractable Organics	NELAP	2/4/2002
Fluorene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Fluorene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Fluorene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Fluoride	EPA 340.2	General Chemistry	NELAP	2/4/2002
Fluoride	SM 4500 F-C	General Chemistry	NELAP	2/4/2002
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
gamma-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
gamma-Chlordane	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
gamma-Chlordane	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Gasoline range organics (GRO)	EPA 8015	Extractable Organics	NELAP	7/1/2003
Gasoline range organics (GRO)	MA-VPH	Extractable Organics	NELAP	7/1/2003
Gasoline range organics (GRO)	MEDEP 4.2.17	Extractable Organics	NELAP	7/1/2003
Hardness	SM 2340 B	Metals	NELAP	2/4/2002
Hardness (calc.)	EPA 200.7	Metals	NELAP	4/26/2002
Heptachlor	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Heptachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Heptachlor	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Heptachlor	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Heptachlor epoxide	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Heptachlor epoxide	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Heptachlor epoxide	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Heptachlor epoxide	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Hexachlorobenzene	EPA 625	Extractable Organics	NELAP	2/4/2002
Hexachlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Hexachlorobenzene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Hexachlorobenzene	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Hexachlorobutadiene	EPA 625	Extractable Organics	NELAP	2/4/2002
Hexachlorobutadiene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Hexachlorobutadiene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Hexachlorobutadiene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Hexachlorobutadiene	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Hexachlorocyclopentadiene	EPA 625	Extractable Organics	NELAP	2/4/2002
Hexachlorocyclopentadiene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Hexachlorocyclopentadiene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Hexachlorocyclopentadiene	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Hexachloroethane	EPA 625	Extractable Organics	NELAP	2/4/2002
Hexachloroethane	EPA 8270	Extractable Organics	NELAP	7/1/2003
Hexachloroethane	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Hexachloroethane	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Hexachloropropene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Ignitability	EPA 1010	General Chemistry	NELAP	7/1/2003
Indeno(1,2,3-cd)pyrene	EPA 625	Extractable Organics	NELAP	2/4/2002
Indeno(1,2,3-cd)pyrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Indeno(1,2,3-cd)pyrene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Indeno(1,2,3-cd)pyrene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Iodomethane (Methyl iodide)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Iron	EPA 200.7	Metals	NELAP	2/4/2002
Iron	EPA 6010	Metals	NELAP	7/1/2003
Iron	EPA 6020	Metals	NELAP	11/7/2006
Iron	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Iron	SM 3500-Fe D (18th/19th Ed.)/UV-VIS	General Chemistry	NELAP	4/26/2002
Isobutyl alcohol (2-Methyl-1-propanol)	EPA 8015	Volatile Organics	NELAP	7/1/2003
Isobutyl alcohol (2-Methyl-1-propanol)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Isodrin	EPA 8270	Extractable Organics	NELAP	7/1/2003

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Isophorone	EPA 625	Extractable Organics	NELAP	2/4/2002
Isophorone	EPA 8270	Extractable Organics	NELAP	7/1/2003
Isophorone	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Isophorone	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Isopropyl alcohol (2-Propanol)	EPA 8015	Volatile Organics	NELAP	7/1/2003
Isopropylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Isopropylbenzene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Isopropylbenzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Isopropylbenzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Isosafrole	EPA 8270	Extractable Organics	NELAP	7/1/2003
Kjeldahl nitrogen - total	EPA 351.2	General Chemistry	NELAP	2/4/2002
Lead	EPA 200.7	Metals	NELAP	2/4/2002
Lead	EPA 200.8	Metals	NELAP	2/4/2002
Lead	EPA 6010	Metals	NELAP	7/1/2003
Lead	EPA 6020	Metals	NELAP	2/4/2002
Lead	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Lead	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006
m+p-Xylenes	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
m+p-Xylenes	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Magnesium	EPA 200.7	Metals	NELAP	2/4/2002
Magnesium	EPA 6010	Metals	NELAP	7/1/2003
Magnesium	EPA 6020	Metals	NELAP	11/7/2006
Magnesium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Magnesium	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006
Manganese	EPA 200.7	Metals	NELAP	2/4/2002
Manganese	EPA 200.8	Metals	NELAP	2/4/2002
Manganese	EPA 6010	Metals	NELAP	7/1/2003
Manganese	EPA 6020	Metals	NELAP	7/1/2003
Manganese	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
MCPA	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
MCPP	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003

Laboratory Scope of Accreditation

Attachment to Certificate #: E87604-13, expiration date June 30, 2010. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: **E87604** EPA Lab Code: **ME00019** **(207) 874-2400**

E87604
Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Mercury	EPA 1631	Metals	NELAP	4/26/2002
Mercury	EPA 245.1	Metals	NELAP	2/4/2002
Mercury	EPA 7470	Metals	NELAP	7/1/2003
Methacrylonitrile	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methanol	EPA 8015	Volatile Organics	NELAP	7/1/2003
Methapyrilene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Methoxychlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Methoxychlor	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Methoxychlor	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Methyl acetate	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Methyl acetate	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methyl acetate	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methyl bromide (Bromomethane)	EPA 624	Volatile Organics	NELAP	2/4/2002
Methyl bromide (Bromomethane)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methyl bromide (Bromomethane)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methyl bromide (Bromomethane)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methyl chloride (Chloromethane)	EPA 624	Volatile Organics	NELAP	2/4/2002
Methyl chloride (Chloromethane)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methyl chloride (Chloromethane)	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Methyl chloride (Chloromethane)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methyl chloride (Chloromethane)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methyl methacrylate	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methyl methanesulfonate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Methyl parathion (Parathion, methyl)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Methyl tert-butyl ether (MTBE)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methyl tert-butyl ether (MTBE)	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Methylcyclohexane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Methylcyclohexane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methylcyclohexane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009

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Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Methylene chloride	EPA 624	Volatile Organics	NELAP	2/4/2002
Methylene chloride	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methylene chloride	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Methylene chloride	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methylene chloride	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Molybdenum	EPA 200.7	Metals	NELAP	2/4/2002
Molybdenum	EPA 200.8	Metals	NELAP	2/4/2002
Molybdenum	EPA 6010	Metals	NELAP	7/1/2003
Molybdenum	EPA 6020	Metals	NELAP	4/26/2002
Naphthalene	EPA 625	Extractable Organics	NELAP	2/4/2002
Naphthalene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Naphthalene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Naphthalene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Naphthalene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
n-Butylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Nickel	EPA 200.7	Metals	NELAP	2/4/2002
Nickel	EPA 200.8	Metals	NELAP	2/4/2002
Nickel	EPA 6010	Metals	NELAP	7/1/2003
Nickel	EPA 6020	Metals	NELAP	2/4/2002
Nickel	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Nickel	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006
Nitrate	EPA 9056	General Chemistry	NELAP	7/1/2003
Nitrate as N	EPA 300.0	General Chemistry	NELAP	2/4/2002
Nitrate as N	EPA 353.2	General Chemistry	NELAP	2/4/2002
Nitrate as N	SM 4500-NO3 F	General Chemistry	NELAP	2/4/2002
Nitrate-nitrite	EPA 300.0	General Chemistry	NELAP	2/4/2002
Nitrate-nitrite	EPA 353.2	General Chemistry	NELAP	2/4/2002
Nitrate-nitrite	SM 4500-NO3 F	General Chemistry	NELAP	2/4/2002
Nitrite	EPA 9056	General Chemistry	NELAP	7/1/2003
Nitrite as N	EPA 300.0	General Chemistry	NELAP	2/4/2002
Nitrite as N	EPA 353.2	General Chemistry	NELAP	2/4/2002
Nitrite as N	SM 4500-NO3 F	General Chemistry	NELAP	2/4/2002
Nitrobenzene	EPA 625	Extractable Organics	NELAP	2/4/2002
Nitrobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003

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Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Nitrobenzene	EPA 8330	Extractable Organics	NELAP	7/30/2004
Nitrobenzene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Nitrobenzene	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Nitroglycerin	EPA 8332	Extractable Organics	NELAP	5/12/2005
Nitroquinoline-1-oxide	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosodiethylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosodimethylamine	EPA 625	Extractable Organics	NELAP	2/4/2002
n-Nitrosodimethylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitroso-di-n-butylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosodi-n-propylamine	EPA 625	Extractable Organics	NELAP	2/4/2002
n-Nitrosodi-n-propylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosodi-n-propylamine	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
n-Nitrosodi-n-propylamine	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
n-Nitrosodiphenylamine	EPA 625	Extractable Organics	NELAP	2/4/2002
n-Nitrosodiphenylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosodiphenylamine	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
n-Nitrosodiphenylamine	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
n-Nitrosomethylethylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosomorpholine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosopiperidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosopyrrolidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Propanol	EPA 8015	Volatile Organics	NELAP	7/1/2003
n-Propylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
o,o,o-Triethyl phosphorothioate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	EPA 8330	Extractable Organics	NELAP	7/30/2004
Oil & Grease	EPA 1664A	General Chemistry	NELAP	2/4/2002
Oil & Grease	EPA 9070	General Chemistry	NELAP	7/1/2003
Organic nitrogen	TKN minus AMMONIA	General Chemistry	NELAP	2/4/2002
Orthophosphate as P	EPA 300.0	General Chemistry	NELAP	2/4/2002
Orthophosphate as P	EPA 365.1	General Chemistry	NELAP	11/7/2006
Orthophosphate as P	EPA 365.2	General Chemistry	NELAP	2/4/2002
Orthophosphate as P	EPA 9056	General Chemistry	NELAP	7/1/2003
Orthophosphate as P	SM 4500-P E	General Chemistry	NELAP	2/4/2002
o-Toluidine	EPA 8270	Extractable Organics	NELAP	7/1/2003

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Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
o-Xylene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
o-Xylene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
p-Dioxane	CA-204.07/GC-MS	Extractable Organics	NELAP	11/7/2006
Pentachlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Pentachloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Pentachloronitrobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Pentachlorophenol	EPA 625	Extractable Organics	NELAP	2/4/2002
Pentachlorophenol	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Pentachlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
Pentachlorophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Pentachlorophenol (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Perchlorate	EPA 314.0	General Chemistry	NELAP	7/30/2004
pH	EPA 150.1	General Chemistry	NELAP	2/4/2002
pH	EPA 9040	General Chemistry	NELAP	7/1/2003
pH	SM 4500-H+-B	General Chemistry	NELAP	4/26/2002
Phenacetin	EPA 8270	Extractable Organics	NELAP	7/1/2003
Phenanthrene	EPA 625	Extractable Organics	NELAP	2/4/2002
Phenanthrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Phenanthrene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Phenanthrene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Phenol	EPA 625	Extractable Organics	NELAP	2/4/2002
Phenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
Phenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Phenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Phorate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Phosphorus, total	EPA 365.4	General Chemistry	NELAP	2/4/2002
p-Isopropyltoluene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Potassium	EPA 200.7	Metals	NELAP	2/4/2002
Potassium	EPA 6010	Metals	NELAP	7/1/2003
Potassium	EPA 6020	Metals	NELAP	11/7/2006
Potassium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Pronamide (Kerb)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Propionitrile (Ethyl cyanide)	EPA 8260	Volatile Organics	NELAP	7/1/2003

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Scarborough, ME 04074

Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Pyrene	EPA 625	Extractable Organics	NELAP	2/4/2002
Pyrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Pyrene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Pyrene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Pyridine	EPA 8270	Extractable Organics	NELAP	7/1/2003
RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)	EPA 8330	Extractable Organics	NELAP	7/30/2004
Residue-filterable (TDS)	EPA 160.1	General Chemistry	NELAP	2/4/2002
Residue-filterable (TDS)	SM 2540 C	General Chemistry	NELAP	2/4/2002
Residue-nonfilterable (TSS)	EPA 160.2	General Chemistry	NELAP	2/4/2002
Residue-nonfilterable (TSS)	SM 2540 D	General Chemistry	NELAP	2/4/2002
Residue-settleable	EPA 160.5	General Chemistry	NELAP	2/4/2002
Residue-settleable	SM 2540 F	General Chemistry	NELAP	2/4/2002
Residue-total	EPA 160.3	General Chemistry	NELAP	2/4/2002
Residue-total	SM 2540 B	General Chemistry	NELAP	2/4/2002
Residue-volatile	EPA 160.4	General Chemistry	NELAP	2/4/2002
Safrole	EPA 8270	Extractable Organics	NELAP	7/1/2003
Salinity	SM 2520 B	General Chemistry	NELAP	2/4/2002
sec-Butylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Selenium	EPA 200.7	Metals	NELAP	2/4/2002
Selenium	EPA 200.8	Metals	NELAP	2/4/2002
Selenium	EPA 6010	Metals	NELAP	7/1/2003
Selenium	EPA 6020	Metals	NELAP	2/4/2002
Selenium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Selenium	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006
Silicon	EPA 200.7	Metals	NELAP	2/4/2002
Silver	EPA 200.7	Metals	NELAP	2/4/2002
Silver	EPA 200.8	Metals	NELAP	2/4/2002
Silver	EPA 6010	Metals	NELAP	7/1/2003
Silver	EPA 6020	Metals	NELAP	7/1/2003
Silver	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Silver	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006
Silvex (2,4,5-TP)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Sodium	EPA 200.7	Metals	NELAP	2/4/2002
Sodium	EPA 6010	Metals	NELAP	7/1/2003
Sodium	EPA 6020	Metals	NELAP	11/7/2006

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Sodium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Strontium	CA-627-02(EPA6020)/ICP-MS	Metals	NELAP	11/7/2006
Strontium	EPA 6010	Metals	NELAP	7/1/2003
Styrene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Styrene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Styrene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Styrene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Sulfate	ASTM D516-02	General Chemistry	NELAP	4/17/2007
Sulfate	ASTM D516-90	General Chemistry	NELAP	4/17/2007
Sulfate	EPA 300.0	General Chemistry	NELAP	2/4/2002
Sulfate	EPA 375.4	General Chemistry	NELAP	2/4/2002
Sulfate	EPA 9038	General Chemistry	NELAP	7/1/2003
Sulfate	EPA 9056	General Chemistry	NELAP	7/1/2003
Sulfide	EPA 376.1	General Chemistry	NELAP	2/4/2002
Sulfide	SM 4500-S E (18th Ed.)/TITR	General Chemistry	NELAP	4/17/2007
Sulfite-SO3	EPA 377.1	General Chemistry	NELAP	2/4/2002
Sulfite-SO3	SM 4500-SO3 B	General Chemistry	NELAP	4/17/2007
Sulfotepp	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Surfactants - MBAS	SM 5540 C	General Chemistry	NELAP	5/12/2005
T-amylmethylether (TAME)	EPA 8260	Volatile Organics	NELAP	5/8/2009
tert-Butyl alcohol	EPA 8260	Volatile Organics	NELAP	5/8/2009
tert-Butylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Tetrachloroethylene (Perchloroethylene)	EPA 624	Volatile Organics	NELAP	2/4/2002
Tetrachloroethylene (Perchloroethylene)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Tetrachloroethylene (Perchloroethylene)	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Tetrachloroethylene (Perchloroethylene)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Tetrachloroethylene (Perchloroethylene)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Tetrahydrofuran (THF)	CA-202.08/GC-MS	Volatile Organics	NELAP	11/7/2006
Tetryl (methyl-2,4,6-trinitrophenylnitramine)	EPA 8330	Extractable Organics	NELAP	7/30/2004
Thallium	EPA 200.7	Metals	NELAP	2/4/2002
Thallium	EPA 200.8	Metals	NELAP	2/4/2002
Thallium	EPA 6010	Metals	NELAP	7/1/2003

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Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Thallium	EPA 6020	Metals	NELAP	7/1/2003
Thallium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Thallium	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006
Thionazin (Zinophos)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Thorium	EPA 200.8	Metals	NELAP	2/4/2002
Tin	CA-628-01(EPA 200.8)/ICP-MS	Metals	NELAP	11/7/2006
Tin	EPA 200.7	Metals	NELAP	2/4/2002
Tin	EPA 6010	Metals	NELAP	7/30/2004
Titanium	EPA 200.7	Metals	NELAP	2/4/2002
Titanium	EPA 6010	Metals	NELAP	7/30/2004
Toluene	EPA 624	Volatile Organics	NELAP	2/4/2002
Toluene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Toluene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Toluene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Toluene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Total coliforms	SM 9222 B	Microbiology	NELAP	7/30/2004
Total cyanide	EPA 9012	General Chemistry	NELAP	7/1/2003
Total hardness as CaCO3	CA-628-01(EPA 200.8)/ICP-MS	Metals	NELAP	11/7/2006
Total hardness as CaCO3	EPA 130.2	General Chemistry	NELAP	9/4/2007
Total hardness as CaCO3	SM 2340 C	General Chemistry	NELAP	9/4/2007
Total nitrate-nitrite	EPA 9056	General Chemistry	NELAP	7/1/2003
Total organic carbon	EPA 415.1	General Chemistry	NELAP	2/4/2002
Total organic carbon	EPA 9060	General Chemistry	NELAP	7/1/2003
Total organic carbon	SM 5310B	General Chemistry	NELAP	4/17/2007
Total Petroleum Hydrocarbons (TPH)	EPA 1664A	General Chemistry	NELAP	2/4/2002
Total Petroleum Hydrocarbons (TPH)	FL-PRO	Extractable Organics	NELAP	7/1/2003
Total Petroleum Hydrocarbons (TPH)	TX1005	Extractable Organics	NELAP	9/4/2007
Total phenolics	EPA 420.1	General Chemistry	NELAP	2/4/2002
Total phenolics	EPA 9065	General Chemistry	NELAP	7/1/2003
Total residual chlorine	SM 4500-Cl G	General Chemistry	NELAP	9/4/2007
Total, fixed, and volatile residue	SM 2540 G	General Chemistry	NELAP	4/26/2002
Toxaphene (Chlorinated camphene)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Toxaphene (Chlorinated camphene)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003

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Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Toxaphene (Chlorinated camphene)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Toxaphene (Chlorinated camphene)	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
trans-1,2-Dichloroethylene	EPA 624	Volatile Organics	NELAP	2/4/2002
trans-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	7/1/2003
trans-1,2-Dichloroethylene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
trans-1,2-Dichloroethylene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
trans-1,2-Dichloroethylene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
trans-1,3-Dichloropropylene	EPA 624	Volatile Organics	NELAP	2/4/2002
trans-1,3-Dichloropropylene	EPA 8260	Volatile Organics	NELAP	7/1/2003
trans-1,3-Dichloropropylene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
trans-1,3-Dichloropropylene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
trans-1,3-Dichloropropylene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
trans-1,4-Dichloro-2-butene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Trichloroethene (Trichloroethylene)	EPA 624	Volatile Organics	NELAP	2/4/2002
Trichloroethene (Trichloroethylene)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Trichloroethene (Trichloroethylene)	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Trichloroethene (Trichloroethylene)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Trichloroethene (Trichloroethylene)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Trichlorofluoromethane	EPA 624	Volatile Organics	NELAP	2/4/2002
Trichlorofluoromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Trichlorofluoromethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Trichlorofluoromethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Trichlorofluoromethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Turbidity	EPA 180.1	General Chemistry	NELAP	2/4/2002
Turbidity	SM 2130 B	General Chemistry	NELAP	2/4/2002
Uranium	EPA 200.8	Metals	NELAP	2/4/2002
Vanadium	EPA 200.7	Metals	NELAP	2/4/2002
Vanadium	EPA 200.8	Metals	NELAP	2/4/2002
Vanadium	EPA 6010	Metals	NELAP	7/1/2003

Laboratory Scope of Accreditation

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State Laboratory ID: **E87604** EPA Lab Code: **ME00019** **(207) 874-2400**

E87604
Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Vanadium	EPA 6020	Metals	NELAP	2/4/2002
Vanadium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Vanadium	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006
Vinyl acetate	EPA 8260	Volatile Organics	NELAP	7/1/2003
Vinyl chloride	EPA 624	Volatile Organics	NELAP	2/4/2002
Vinyl chloride	EPA 8260	Volatile Organics	NELAP	7/1/2003
Vinyl chloride	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Vinyl chloride	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Vinyl chloride	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Xylene (total)	EPA 624	Volatile Organics	NELAP	4/26/2002
Xylene (total)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Xylene (total)	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Zinc	EPA 200.7	Metals	NELAP	2/4/2002
Zinc	EPA 200.8	Metals	NELAP	2/4/2002
Zinc	EPA 6010	Metals	NELAP	7/1/2003
Zinc	EPA 6020	Metals	NELAP	2/4/2002
Zinc	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Zinc	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006

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600 Technology Way
Scarborough, ME 04074

Matrix: **Solid and Chemical Materials**

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,1,1-Trichloroethane	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,1,1-Trichloroethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,1,1-Trichloroethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1,1-Trichloroethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1,2,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,1,2,2-Tetrachloroethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,1,2,2-Tetrachloroethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1,2,2-Tetrachloroethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1,2-Trichloro-1,2,2-trifluoroethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,1,2-Trichloro-1,2,2-trifluoroethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1,2-Trichloro-1,2,2-trifluoroethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1,2-Trichloroethane	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,1,2-Trichloroethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,1,2-Trichloroethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1,2-Trichloroethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1-Dichloroethane	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,1-Dichloroethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,1-Dichloroethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1-Dichloroethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,1-Dichloroethylene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,1-Dichloroethylene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1-Dichloroethylene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1-Dichloropropene	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,2,3-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	2/4/2002

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Matrix: **Solid and Chemical Materials**

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,2,3-Trichlorobenzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2,3-Trichlorobenzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2,3-Trichloropropane	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,2,4,5-Tetrachlorobenzene	EPA 8270	Extractable Organics	NELAP	2/4/2002
1,2,4-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,2,4-Trichlorobenzene	EPA 8270	Extractable Organics	NELAP	2/4/2002
1,2,4-Trichlorobenzene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,2,4-Trichlorobenzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2,4-Trichlorobenzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2,4-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,2-Dibromo-3-chloropropane (DBCP)	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,2-Dibromo-3-chloropropane (DBCP)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dibromo-3-chloropropane (DBCP) (with SIM)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,2-Dibromoethane (EDB, Ethylene dibromide)	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,2-Dibromoethane (EDB, Ethylene dibromide)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dibromoethane (EDB, Ethylene dibromide) (with SIM)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,2-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	2/4/2002
1,2-Dichlorobenzene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,2-Dichlorobenzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dichlorobenzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dichloroethane	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,2-Dichloroethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,2-Dichloroethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dichloroethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009

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Scarborough, ME 04074

Matrix: **Solid and Chemical Materials**

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,2-Dichloropropane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,2-Dichloropropane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dichloropropane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Diphenylhydrazine	EPA 8270	Extractable Organics	NELAP	2/4/2002
1,3,5-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,3,5-Trinitrobenzene (1,3,5-TNB)	EPA 8270	Extractable Organics	NELAP	2/4/2002
1,3,5-Trinitrobenzene (1,3,5-TNB)	EPA 8330	Extractable Organics	NELAP	7/30/2004
1,3-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,3-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	2/4/2002
1,3-Dichlorobenzene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,3-Dichlorobenzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,3-Dichlorobenzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,3-Dichloropropane	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,3-Dinitrobenzene (1,3-DNB)	EPA 8270	Extractable Organics	NELAP	4/26/2002
1,3-Dinitrobenzene (1,3-DNB)	EPA 8330	Extractable Organics	NELAP	7/30/2004
1,4-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,4-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	2/4/2002
1,4-Dichlorobenzene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,4-Dichlorobenzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,4-Dichlorobenzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,4-Dioxane (1,4-Diethyleneoxide)	EPA 8260	Volatile Organics	NELAP	4/26/2002
1,4-Dioxane (1,4-Diethyleneoxide)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,4-Dioxane (1,4-Diethyleneoxide) (without SIM)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,4-Naphthoquinone	EPA 8270	Extractable Organics	NELAP	2/4/2002
1,4-Phenylenediamine	EPA 8270	Extractable Organics	NELAP	2/4/2002
1-Naphthylamine	EPA 8270	Extractable Organics	NELAP	2/4/2002
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (BZ 206)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',3,3',4,4',5,6-Octachlorobiphenyl (BZ 195)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ 170)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004

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600 Technology Way
Scarborough, ME 04074

Matrix: **Solid and Chemical Materials**

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,2',3,3',4,4'-Hexachlorobiphenyl (BZ 128)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,2',3,4,4',5,5'-Heptachlorobiphenyl (BZ 180)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',3,4,4',5',6-Heptachlorobiphenyl (BZ 183)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',3,4,4',5'-Hexachlorobiphenyl (BZ 138)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',3,4,4',6,6'-Heptachlorobiphenyl (BZ 184)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,2',3,4',5,5',6-Heptachlorobiphenyl (BZ 187)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',3,4,5'-Pentachlorobiphenyl (BZ 87)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',3,5'-Tetrachlorobiphenyl (BZ 44)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',4,4',5,5'-Hexachlorobiphenyl (BZ 153)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',4,5,5'-Pentachlorobiphenyl (BZ 101)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',4,5'-Tetrachlorobiphenyl (BZ 49)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,2',5,5'-Tetrachlorobiphenyl (BZ 52)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',5-Trichlorobiphenyl (BZ 18)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	2/4/2002
2,3,3',4,4',5,5'-Heptachlorobiphenyl (BZ 189)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,3,3',4,4',5-Hexachlorobiphenyl (BZ 156)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,3,3',4,4',5'-Hexachlorobiphenyl (BZ 157)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,3,3',4,4'-Pentachlorobiphenyl (BZ 105)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,3',4,4',5,5'-Hexachlorobiphenyl (BZ 167)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,3,4,4',5-Pentachlorobiphenyl (BZ 114)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,3',4,4',5-Pentachlorobiphenyl (BZ 118)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,3',4,4',5'-Pentachlorobiphenyl (BZ 123)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,3',4,4'-Tetrachlorobiphenyl (BZ 66)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,3,4,6-Tetrachlorophenol	EPA 8270	Extractable Organics	NELAP	2/4/2002
2,3,4,6-Tetrachlorophenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2,4,4'-Trichlorobiphenyl (BZ 28)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,4,5-T	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	5/12/2005
2,4,5-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	2/4/2002
2,4,5-Trichlorophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2,4,5-Trichlorophenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2,4,6-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	2/4/2002
2,4,6-Trichlorophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2,4,6-Trichlorophenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2,4,6-Trinitrotoluene (2,4,6-TNT)	EPA 8330	Extractable Organics	NELAP	7/30/2004
2,4-D	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	5/12/2005

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Matrix: **Solid and Chemical Materials**

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,4-DB	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	5/12/2005
2,4'-Dichlorobiphenyl (BZ 8)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,4-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	2/4/2002
2,4-Dichlorophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2,4-Dichlorophenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2,4-Dimethylphenol	EPA 8270	Extractable Organics	NELAP	2/4/2002
2,4-Dimethylphenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2,4-Dimethylphenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2,4-Dinitrophenol	EPA 8270	Extractable Organics	NELAP	2/4/2002
2,4-Dinitrophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2,4-Dinitrophenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2,4-Dinitrotoluene (2,4-DNT)	EPA 8270	Extractable Organics	NELAP	2/4/2002
2,4-Dinitrotoluene (2,4-DNT)	EPA 8330	Extractable Organics	NELAP	7/30/2004
2,4-Dinitrotoluene (2,4-DNT)	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2,4-Dinitrotoluene (2,4-DNT)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2,6-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	2/4/2002
2,6-Dinitrotoluene (2,6-DNT)	EPA 8270	Extractable Organics	NELAP	2/4/2002
2,6-Dinitrotoluene (2,6-DNT)	EPA 8330	Extractable Organics	NELAP	7/30/2004
2,6-Dinitrotoluene (2,6-DNT)	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2-Acetylaminofluorene	EPA 8270	Extractable Organics	NELAP	2/4/2002
2-Amino-4,6-dinitrotoluene (2-am-dnt)	EPA 8330	Extractable Organics	NELAP	7/30/2004
2-Butanone (Methyl ethyl ketone, MEK)	EPA 8260	Volatile Organics	NELAP	2/4/2002
2-Butanone (Methyl ethyl ketone, MEK)	OLM04.3-Exhibit D	Volatile Organics	NELAP	5/12/2005
2-Butanone (Methyl ethyl ketone, MEK)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
2-Butanone (Methyl ethyl ketone, MEK)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
2-Chloroethyl vinyl ether	EPA 8260	Volatile Organics	NELAP	2/4/2002
2-Chloronaphthalene	EPA 8270	Extractable Organics	NELAP	2/4/2002
2-Chloronaphthalene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2-Chloronaphthalene	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2-Chlorophenol	EPA 8270	Extractable Organics	NELAP	2/4/2002
2-Chlorophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2-Chlorophenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009

Laboratory Scope of Accreditation

Attachment to Certificate #: E87604-13, expiration date June 30, 2010. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: **E87604** EPA Lab Code: **ME00019** **(207) 874-2400**

E87604
Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: **Solid and Chemical Materials**

Analyte	Method/Tech	Category	Certification Type	Effective Date
2-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	2/4/2002
2-Hexanone	EPA 8260	Volatile Organics	NELAP	2/4/2002
2-Hexanone	OLM04.3-Exhibit D	Volatile Organics	NELAP	5/12/2005
2-Hexanone	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
2-Hexanone	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
2-Methyl-4,6-dinitrophenol	EPA 8270	Extractable Organics	NELAP	2/4/2002
2-Methyl-4,6-dinitrophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2-Methylnaphthalene	EPA 8270	Extractable Organics	NELAP	2/4/2002
2-Methylnaphthalene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2-Methylnaphthalene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2-Methylphenol (o-Cresol)	EPA 8270	Extractable Organics	NELAP	2/4/2002
2-Methylphenol (o-Cresol)	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2-Methylphenol (o-Cresol)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2-Naphthylamine	EPA 8270	Extractable Organics	NELAP	2/4/2002
2-Nitroaniline	EPA 8270	Extractable Organics	NELAP	2/4/2002
2-Nitroaniline	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2-Nitrophenol	EPA 8270	Extractable Organics	NELAP	2/4/2002
2-Nitrophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2-Nitrophenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	7/30/2004
2-Picoline (2-Methylpyridine)	EPA 8270	Extractable Organics	NELAP	2/4/2002
3,3',4,4',5,5'-Hexachlorobiphenyl (BZ 169)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
3,3',4,4',5-Pentachlorobiphenyl (BZ 126)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
3,3',4,4'-Tetrachlorobiphenyl (BZ 77)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
3,3'-Dichlorobenzidine	EPA 8270	Extractable Organics	NELAP	2/4/2002
3,3'-Dichlorobenzidine	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
3,3'-Dichlorobenzidine	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
3,3'-Dimethylbenzidine	EPA 8270	Extractable Organics	NELAP	2/4/2002
3,4,4',5-Tetrachlorobiphenyl (BZ 81)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
3-Methylcholanthrene	EPA 8270	Extractable Organics	NELAP	4/26/2002
3-Nitroaniline	EPA 8270	Extractable Organics	NELAP	2/4/2002
3-Nitroaniline	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004

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Katahdin Analytical Services, Inc.
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Scarborough, ME 04074

Matrix: **Solid and Chemical Materials**

Analyte	Method/Tech	Category	Certification Type	Effective Date
3-Nitroaniline	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
3-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	7/30/2004
4,4'-DDD	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
4,4'-DDD	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
4,4'-DDD	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
4,4'-DDE	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
4,4'-DDE	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
4,4'-DDE	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
4,4'-DDT	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
4,4'-DDT	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
4,4'-DDT	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
4-Amino-2,6-dinitrotoluene (4-am-dnt)	EPA 8330	Extractable Organics	NELAP	7/30/2004
4-Aminobiphenyl	EPA 8270	Extractable Organics	NELAP	2/4/2002
4-Bromophenyl phenyl ether	EPA 8270	Extractable Organics	NELAP	2/4/2002
4-Bromophenyl phenyl ether	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
4-Bromophenyl phenyl ether	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
4-Chloro-3-methylphenol	EPA 8270	Extractable Organics	NELAP	2/4/2002
4-Chloro-3-methylphenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
4-Chloro-3-methylphenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
4-Chloroaniline	EPA 8270	Extractable Organics	NELAP	2/4/2002
4-Chloroaniline	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
4-Chloroaniline	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
4-Chlorophenyl phenylether	EPA 8270	Extractable Organics	NELAP	2/4/2002
4-Chlorophenyl phenylether	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
4-Chlorophenyl phenylether	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
4-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	2/4/2002
4-Dimethyl aminoazobenzene	EPA 8270	Extractable Organics	NELAP	2/4/2002
4-Methyl-2-pentanone (MIBK)	EPA 8260	Volatile Organics	NELAP	2/4/2002
4-Methyl-2-pentanone (MIBK)	OLM04.3-Exhibit D	Volatile Organics	NELAP	5/12/2005
4-Methyl-2-pentanone (MIBK)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
4-Methyl-2-pentanone (MIBK)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009

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Scarborough, ME 04074

Matrix: **Solid and Chemical Materials**

Analyte	Method/Tech	Category	Certification Type	Effective Date
4-Methylphenol (p-Cresol)	EPA 8270	Extractable Organics	NELAP	2/4/2002
4-Methylphenol (p-Cresol)	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
4-Methylphenol (p-Cresol)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
4-Nitroaniline	EPA 8270	Extractable Organics	NELAP	2/4/2002
4-Nitroaniline	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
4-Nitroaniline	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
4-Nitrophenol	EPA 8270	Extractable Organics	NELAP	2/4/2002
4-Nitrophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
4-Nitrophenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
4-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	7/30/2004
5-Nitro-o-toluidine	EPA 8270	Extractable Organics	NELAP	2/4/2002
7,12-Dimethylbenz(a) anthracene	EPA 8270	Extractable Organics	NELAP	2/4/2002
a-a-Dimethylphenethylamine	EPA 8270	Extractable Organics	NELAP	2/4/2002
Acenaphthene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Acenaphthene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Acenaphthene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Acenaphthylene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Acenaphthylene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Acenaphthylene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Acetone	EPA 8260	Volatile Organics	NELAP	2/4/2002
Acetone	OLM04.3-Exhibit D	Volatile Organics	NELAP	5/12/2005
Acetone	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Acetone	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Acetonitrile	EPA 8260	Volatile Organics	NELAP	2/4/2002
Acetophenone	EPA 8270	Extractable Organics	NELAP	2/4/2002
Acetophenone	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Acrolein (Propenal)	EPA 8260	Volatile Organics	NELAP	2/4/2002
Acrylonitrile	EPA 8260	Volatile Organics	NELAP	2/4/2002
Aldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Aldrin	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aldrin	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Allyl chloride (3-Chloropropene)	EPA 8260	Volatile Organics	NELAP	4/26/2002

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Analyte	Method/Tech	Category	Certification Type	Effective Date
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
alpha-BHC (alpha-Hexachlorocyclohexane)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
alpha-BHC (alpha-Hexachlorocyclohexane)	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
alpha-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/26/2002
alpha-Chlordane	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
alpha-Chlordane	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aluminum	EPA 6010	Metals	NELAP	2/4/2002
Aluminum	EPA 6020	Metals	NELAP	2/4/2002
Aluminum	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Amenable cyanide	EPA 9012	General Chemistry	NELAP	4/26/2002
Aniline	EPA 8270	Extractable Organics	NELAP	2/4/2002
Anthracene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Anthracene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Anthracene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Antimony	EPA 6010	Metals	NELAP	2/4/2002
Antimony	EPA 6020	Metals	NELAP	2/4/2002
Antimony	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Aramite	EPA 8270	Extractable Organics	NELAP	2/4/2002
Aroclor-1016 (PCB-1016)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Aroclor-1016 (PCB-1016)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1016 (PCB-1016)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aroclor-1221 (PCB-1221)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Aroclor-1221 (PCB-1221)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1221 (PCB-1221)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aroclor-1232 (PCB-1232)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Aroclor-1232 (PCB-1232)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1232 (PCB-1232)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aroclor-1242 (PCB-1242)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Aroclor-1242 (PCB-1242)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1242 (PCB-1242)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aroclor-1248 (PCB-1248)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Aroclor-1248 (PCB-1248)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004

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Scarborough, ME 04074

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Aroclor-1248 (PCB-1248)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aroclor-1254 (PCB-1254)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Aroclor-1254 (PCB-1254)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1254 (PCB-1254)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aroclor-1260 (PCB-1260)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Aroclor-1260 (PCB-1260)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1260 (PCB-1260)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aroclor-1262 (PCB-1262)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aroclor-1268 (PCB-1268)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Arsenic	EPA 6010	Metals	NELAP	2/4/2002
Arsenic	EPA 6020	Metals	NELAP	2/4/2002
Arsenic	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Atrazine	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Atrazine	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Barium	EPA 6010	Metals	NELAP	2/4/2002
Barium	EPA 6020	Metals	NELAP	2/4/2002
Barium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Benzaldehyde	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Benzaldehyde	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Benzene	EPA 8260	Volatile Organics	NELAP	2/4/2002
Benzene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Benzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Benzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Benzidine	EPA 8270	Extractable Organics	NELAP	2/4/2002
Benzo(a)anthracene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Benzo(a)anthracene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Benzo(a)anthracene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Benzo(a)pyrene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Benzo(a)pyrene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Benzo(a)pyrene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009

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Benzo(b)fluoranthene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Benzo(b)fluoranthene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Benzo(b)fluoranthene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Benzo(g,h,i)perylene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Benzo(g,h,i)perylene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Benzo(g,h,i)perylene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Benzo(k)fluoranthene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Benzo(k)fluoranthene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Benzo(k)fluoranthene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Benzoic acid	EPA 8270	Extractable Organics	NELAP	2/4/2002
Benzyl alcohol	EPA 8270	Extractable Organics	NELAP	2/4/2002
Beryllium	EPA 6010	Metals	NELAP	2/4/2002
Beryllium	EPA 6020	Metals	NELAP	2/4/2002
Beryllium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
beta-BHC (beta-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
beta-BHC (beta-Hexachlorocyclohexane)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
beta-BHC (beta-Hexachlorocyclohexane)	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Biphenyl	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Biphenyl	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
bis(2-Chloroethoxy)methane	EPA 8270	Extractable Organics	NELAP	2/4/2002
bis(2-Chloroethoxy)methane	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
bis(2-Chloroethoxy)methane	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
bis(2-Chloroethyl) ether	EPA 8270	Extractable Organics	NELAP	2/4/2002
bis(2-Chloroethyl) ether	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
bis(2-Chloroethyl) ether	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 8270	Extractable Organics	NELAP	2/4/2002
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 8270	Extractable Organics	NELAP	2/4/2002
bis(2-Ethylhexyl) phthalate (DEHP)	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
bis(2-Ethylhexyl) phthalate (DEHP)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009

Laboratory Scope of Accreditation

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State Laboratory ID: **E87604** EPA Lab Code: **ME00019** **(207) 874-2400**

E87604
Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: **Solid and Chemical Materials**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Boron	CA-627-02(EPA6020)/ICP-MS	Metals	NELAP	11/7/2006
Boron	EPA 6010	Metals	NELAP	4/26/2002
Bromide	EPA 9056	General Chemistry	NELAP	2/4/2002
Bromobenzene	EPA 8260	Volatile Organics	NELAP	2/4/2002
Bromochloromethane	EPA 8260	Volatile Organics	NELAP	2/4/2002
Bromochloromethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Bromochloromethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Bromodichloromethane	EPA 8260	Volatile Organics	NELAP	2/4/2002
Bromodichloromethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Bromodichloromethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Bromodichloromethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Bromoform	EPA 8260	Volatile Organics	NELAP	2/4/2002
Bromoform	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Bromoform	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Bromoform	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Butyl benzyl phthalate	EPA 8270	Extractable Organics	NELAP	2/4/2002
Butyl benzyl phthalate	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Butyl benzyl phthalate	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Cadmium	EPA 6010	Metals	NELAP	2/4/2002
Cadmium	EPA 6020	Metals	NELAP	2/4/2002
Cadmium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Calcium	EPA 6010	Metals	NELAP	2/4/2002
Calcium	EPA 6020	General Chemistry	NELAP	11/7/2006
Calcium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Caprolactam	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Caprolactam	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Carbazole	EPA 8270	Extractable Organics	NELAP	2/4/2002
Carbazole	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Carbazole	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009

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Scarborough, ME 04074

Matrix: **Solid and Chemical Materials**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Carbon disulfide	EPA 8260	Volatile Organics	NELAP	2/4/2002
Carbon disulfide	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Carbon disulfide	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Carbon disulfide	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Carbon tetrachloride	EPA 8260	Volatile Organics	NELAP	2/4/2002
Carbon tetrachloride	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Carbon tetrachloride	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Carbon tetrachloride	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Chlordane (tech.)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Chloride	EPA 9056	General Chemistry	NELAP	2/4/2002
Chloride	EPA 9251	General Chemistry	NELAP	2/4/2002
Chlorobenzene	EPA 8260	Volatile Organics	NELAP	2/4/2002
Chlorobenzene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Chlorobenzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Chlorobenzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Chlorobenzilate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Chloroethane	EPA 8260	Volatile Organics	NELAP	2/4/2002
Chloroethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Chloroethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Chloroethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Chloroform	EPA 8260	Volatile Organics	NELAP	2/4/2002
Chloroform	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Chloroform	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Chloroform	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Chloroprene	EPA 8260	Volatile Organics	NELAP	2/4/2002
Chromium	EPA 6010	Metals	NELAP	2/4/2002
Chromium	EPA 6020	Metals	NELAP	2/4/2002
Chromium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006

Laboratory Scope of Accreditation

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Katahdin Analytical Services, Inc.
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Scarborough, ME 04074

Matrix: **Solid and Chemical Materials**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Chromium VI	EPA 7196	General Chemistry	NELAP	4/26/2002
Chrysene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Chrysene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Chrysene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
cis-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	2/4/2002
cis-1,2-Dichloroethylene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
cis-1,2-Dichloroethylene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
cis-1,2-Dichloroethylene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
cis-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	2/4/2002
cis-1,3-Dichloropropene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
cis-1,3-Dichloropropene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
cis-1,3-Dichloropropene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Cobalt	EPA 6010	Metals	NELAP	2/4/2002
Cobalt	EPA 6020	Metals	NELAP	2/4/2002
Cobalt	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Copper	EPA 6010	Metals	NELAP	2/4/2002
Copper	EPA 6020	Metals	NELAP	2/4/2002
Copper	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Cyclohexane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Cyclohexane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Cyclohexane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Dalapon	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	5/12/2005
Decachlorobiphenyl (BZ 209)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
delta-BHC	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
delta-BHC	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
delta-BHC	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Diallate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Dibenz(a,h)anthracene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Dibenz(a,h)anthracene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Dibenz(a,h)anthracene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Dibenzofuran	EPA 8270	Extractable Organics	NELAP	2/4/2002
Dibenzofuran	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Dibromochloromethane	EPA 8260	Volatile Organics	NELAP	2/4/2002
Dibromochloromethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Dibromochloromethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Dibromochloromethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Dibromomethane	EPA 8260	Volatile Organics	NELAP	2/4/2002
Dicamba	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	5/12/2005
Dichlorodifluoromethane	EPA 8260	Volatile Organics	NELAP	2/4/2002
Dichlorodifluoromethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Dichlorodifluoromethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Dichlorodifluoromethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Dichloroprop (Dichlorprop)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	5/12/2005
Dieldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Dieldrin	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Dieldrin	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Diesel range organics (DRO)	EPA 8015	Extractable Organics	NELAP	2/4/2002
Diesel range organics (DRO)	MA-EPH	Extractable Organics	NELAP	2/4/2002
Diesel range organics (DRO)	MEDEP 4.1.25	Extractable Organics	NELAP	2/4/2002
Diethyl ether	EPA 8260	Volatile Organics	NELAP	2/4/2002
Diethyl phthalate	EPA 8270	Extractable Organics	NELAP	2/4/2002
Diethyl phthalate	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Diethyl phthalate	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Di-isopropylether (DIPE)	EPA 8260	Extractable Organics	NELAP	5/8/2009
Dimethoate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	4/26/2002
Dimethyl phthalate	EPA 8270	Extractable Organics	NELAP	2/4/2002
Dimethyl phthalate	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Di-n-butyl phthalate	EPA 8270	Extractable Organics	NELAP	2/4/2002
Di-n-butyl phthalate	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Di-n-butyl phthalate	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009

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Matrix: **Solid and Chemical Materials**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Di-n-octyl phthalate	EPA 8270	Extractable Organics	NELAP	2/4/2002
Di-n-octyl phthalate	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Di-n-octyl phthalate	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	5/12/2005
Endosulfan I	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Endosulfan I	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endosulfan I	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Endosulfan II	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Endosulfan II	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endosulfan II	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Endosulfan sulfate	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Endosulfan sulfate	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endosulfan sulfate	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Endrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Endrin	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endrin	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Endrin aldehyde	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Endrin aldehyde	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endrin aldehyde	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Endrin ketone	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Endrin ketone	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endrin ketone	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Ethyl methacrylate	EPA 8260	Volatile Organics	NELAP	2/4/2002
Ethyl methanesulfonate	EPA 8270	Extractable Organics	NELAP	2/4/2002
Ethylbenzene	EPA 8260	Volatile Organics	NELAP	2/4/2002
Ethylbenzene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Ethylbenzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Ethylbenzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Ethyl-t-butylether (ETBE)	EPA 8260	Extractable Organics	NELAP	5/8/2009
Famphur	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	4/26/2002
Fluoranthene	EPA 8270	Extractable Organics	NELAP	2/4/2002

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Fluoranthene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Fluoranthene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Fluorene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Fluorene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Fluorene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
gamma-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/26/2002
gamma-Chlordane	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
gamma-Chlordane	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Gasoline range organics (GRO)	EPA 8015	Extractable Organics	NELAP	2/4/2002
Gasoline range organics (GRO)	MA-VPH	Extractable Organics	NELAP	2/4/2002
Gasoline range organics (GRO)	MEDEP 4.2.17	Extractable Organics	NELAP	2/4/2002
Heptachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Heptachlor	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Heptachlor	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Heptachlor epoxide	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Heptachlor epoxide	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Heptachlor epoxide	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Hexachlorobenzene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Hexachlorobenzene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Hexachlorobenzene	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Hexachlorobutadiene	EPA 8260	Volatile Organics	NELAP	2/4/2002
Hexachlorobutadiene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Hexachlorobutadiene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Hexachlorobutadiene	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Hexachlorocyclopentadiene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Hexachlorocyclopentadiene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Hexachlorocyclopentadiene	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Hexachloroethane	EPA 8270	Extractable Organics	NELAP	2/4/2002

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Hexachloroethane	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Hexachloroethane	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Hexachloropropene	EPA 8270	Extractable Organics	NELAP	4/26/2002
Ignitability	EPA 1010	General Chemistry	NELAP	2/4/2002
Indeno(1,2,3-cd)pyrene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Indeno(1,2,3-cd)pyrene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Indeno(1,2,3-cd)pyrene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Iodomethane (Methyl iodide)	EPA 8260	Volatile Organics	NELAP	2/4/2002
Iron	EPA 6010	Metals	NELAP	2/4/2002
Iron	EPA 6020	General Chemistry	NELAP	11/7/2006
Iron	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Isobutyl alcohol (2-Methyl-1-propanol)	EPA 8260	Volatile Organics	NELAP	2/4/2002
Isodrin	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Isophorone	EPA 8270	Extractable Organics	NELAP	2/4/2002
Isophorone	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Isophorone	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Isopropylbenzene	EPA 8260	Volatile Organics	NELAP	2/4/2002
Isopropylbenzene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Isopropylbenzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Isopropylbenzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Isosafrole	EPA 8270	Extractable Organics	NELAP	2/4/2002
Lead	EPA 6010	Metals	NELAP	2/4/2002
Lead	EPA 6020	Metals	NELAP	2/4/2002
Lead	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
m+p-Xylenes	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
m+p-Xylenes	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Magnesium	EPA 6010	Metals	NELAP	2/4/2002
Magnesium	EPA 6020	General Chemistry	NELAP	11/7/2006
Magnesium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Manganese	EPA 6010	Metals	NELAP	2/4/2002

Laboratory Scope of Accreditation

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State Laboratory ID: **E87604** EPA Lab Code: **ME00019** **(207) 874-2400**

E87604
Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: **Solid and Chemical Materials**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Manganese	EPA 6020	Metals	NELAP	2/4/2002
Manganese	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
MCPA	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	5/12/2005
MCPP	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	5/12/2005
Mercury	EPA 7471	Metals	NELAP	2/4/2002
Methacrylonitrile	EPA 8260	Volatile Organics	NELAP	2/4/2002
Methapyrilene	EPA 8270	Extractable Organics	NELAP	4/26/2002
Methoxychlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Methoxychlor	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Methoxychlor	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Methyl acetate	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Methyl acetate	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methyl acetate	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methyl bromide (Bromomethane)	EPA 8260	Volatile Organics	NELAP	2/4/2002
Methyl bromide (Bromomethane)	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Methyl bromide (Bromomethane)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methyl bromide (Bromomethane)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methyl chloride (Chloromethane)	EPA 8260	Volatile Organics	NELAP	2/4/2002
Methyl chloride (Chloromethane)	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Methyl chloride (Chloromethane)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methyl chloride (Chloromethane)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methyl methacrylate	EPA 8260	Volatile Organics	NELAP	2/4/2002
Methyl methanesulfonate	EPA 8270	Extractable Organics	NELAP	2/4/2002
Methyl parathion (Parathion, methyl)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	4/26/2002
Methyl tert-butyl ether (MTBE)	EPA 8260	Volatile Organics	NELAP	2/4/2002
Methyl tert-butyl ether (MTBE)	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Methyl tert-butyl ether (MTBE)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methyl tert-butyl ether (MTBE)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methylcyclohexane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004

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Matrix: **Solid and Chemical Materials**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Methylcyclohexane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methylcyclohexane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methylene chloride	EPA 8260	Volatile Organics	NELAP	2/4/2002
Methylene chloride	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Methylene chloride	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methylene chloride	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Molybdenum	EPA 6010	Metals	NELAP	4/26/2002
Molybdenum	EPA 6020	General Chemistry	NELAP	11/7/2006
Naphthalene	EPA 8260	Volatile Organics	NELAP	2/4/2002
Naphthalene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Naphthalene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Naphthalene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
n-Butylbenzene	EPA 8260	Volatile Organics	NELAP	2/4/2002
Nickel	EPA 6010	Metals	NELAP	2/4/2002
Nickel	EPA 6020	Metals	NELAP	2/4/2002
Nickel	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Nitrate	EPA 9056	General Chemistry	NELAP	2/4/2002
Nitrite	EPA 9056	General Chemistry	NELAP	2/4/2002
Nitrobenzene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Nitrobenzene	EPA 8330	Extractable Organics	NELAP	7/30/2004
Nitrobenzene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Nitrobenzene	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Nitroglycerin	EPA 8332	Extractable Organics	NELAP	5/12/2005
Nitroquinoline-1-oxide	EPA 8270	Extractable Organics	NELAP	2/4/2002
n-Nitrosodiethylamine	EPA 8270	Extractable Organics	NELAP	2/4/2002
n-Nitrosodimethylamine	EPA 8270	Extractable Organics	NELAP	2/4/2002
n-Nitroso-di-n-butylamine	EPA 8270	Extractable Organics	NELAP	4/26/2002
n-Nitrosodi-n-propylamine	EPA 8270	Extractable Organics	NELAP	2/4/2002
n-Nitrosodi-n-propylamine	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
n-Nitrosodi-n-propylamine	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
n-Nitrosodiphenylamine	EPA 8270	Extractable Organics	NELAP	2/4/2002

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Scarborough, ME 04074

Matrix: **Solid and Chemical Materials**

Analyte	Method/Tech	Category	Certification Type	Effective Date
n-Nitrosodiphenylamine	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
n-Nitrosomethylethylamine	EPA 8270	Extractable Organics	NELAP	2/4/2002
n-Nitrosomorpholine	EPA 8270	Extractable Organics	NELAP	2/4/2002
n-Nitrosopiperidine	EPA 8270	Extractable Organics	NELAP	2/4/2002
n-Nitrosopyrrolidine	EPA 8270	Extractable Organics	NELAP	2/4/2002
n-Propylbenzene	EPA 8260	Volatile Organics	NELAP	2/4/2002
o,o,o-Triethyl phosphorothioate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	EPA 8330	Extractable Organics	NELAP	7/30/2004
Oil & Grease	EPA 9071	General Chemistry	NELAP	2/4/2002
Orthophosphate as P	EPA 9056	General Chemistry	NELAP	2/4/2002
o-Toluidine	EPA 8270	Extractable Organics	NELAP	2/4/2002
o-Xylene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
o-Xylene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Paint Filter Liquids Test	EPA 9095	General Chemistry	NELAP	2/4/2002
p-Dioxane	CA-204.07/GC-MS	Extractable Organics	NELAP	11/7/2006
p-Dioxane	EPA 8260	Volatile Organics	NELAP	2/4/2002
Pentachlorobenzene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Pentachloroethane	EPA 8260	Volatile Organics	NELAP	2/4/2002
Pentachloronitrobenzene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Pentachlorophenol	EPA 8270	Extractable Organics	NELAP	2/4/2002
Pentachlorophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Pentachlorophenol (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Perchlorate	EPA 314.0	General Chemistry	NELAP	7/30/2004
pH	EPA 9040	General Chemistry	NELAP	2/4/2002
pH	EPA 9045	General Chemistry	NELAP	2/4/2002
Phenacetin	EPA 8270	Extractable Organics	NELAP	2/4/2002
Phenanthrene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Phenanthrene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Phenanthrene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Phenol	EPA 8270	Extractable Organics	NELAP	2/4/2002
Phenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Phenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Phorate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	4/26/2002

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Matrix: **Solid and Chemical Materials**

Analyte	Method/Tech	Category	Certification Type	Effective Date
p-Isopropyltoluene	EPA 8260	Volatile Organics	NELAP	2/4/2002
Potassium	EPA 6010	Metals	NELAP	2/4/2002
Potassium	EPA 6020	General Chemistry	NELAP	11/7/2006
Potassium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Pronamide (Kerb)	EPA 8270	Extractable Organics	NELAP	2/4/2002
Propionitrile (Ethyl cyanide)	EPA 8260	Volatile Organics	NELAP	2/4/2002
Pyrene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Pyrene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Pyrene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Pyridine	EPA 8270	Extractable Organics	NELAP	2/4/2002
RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)	EPA 8330	Extractable Organics	NELAP	7/30/2004
Safrole	EPA 8270	Extractable Organics	NELAP	2/4/2002
sec-Butylbenzene	EPA 8260	Volatile Organics	NELAP	2/4/2002
Selenium	EPA 6010	Metals	NELAP	2/4/2002
Selenium	EPA 6020	Metals	NELAP	2/4/2002
Selenium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Silver	EPA 6010	Metals	NELAP	2/4/2002
Silver	EPA 6020	Metals	NELAP	2/4/2002
Silver	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Silvex (2,4,5-TP)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	5/12/2005
Sodium	EPA 6010	Metals	NELAP	2/4/2002
Sodium	EPA 6020	General Chemistry	NELAP	11/7/2006
Sodium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Strontium	CA-627-02(EPA6020)/ICP-MS	Metals	NELAP	11/7/2006
Strontium	EPA 6010	Metals	NELAP	2/4/2002
Styrene	EPA 8260	Volatile Organics	NELAP	2/4/2002
Styrene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Styrene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Styrene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Sulfate	EPA 9038	General Chemistry	NELAP	2/4/2002
Sulfate	EPA 9056	General Chemistry	NELAP	2/4/2002
Sulfotepp	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	4/26/2002

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Matrix: **Solid and Chemical Materials**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Synthetic Precipitation Leaching Procedure	EPA 1312	General Chemistry	NELAP	2/4/2002
T-amylmethylether (TAME)	EPA 8260	Extractable Organics	NELAP	5/8/2009
tert-Butyl alcohol	EPA 8260	Extractable Organics	NELAP	5/8/2009
tert-Butylbenzene	EPA 8260	Volatile Organics	NELAP	2/4/2002
Tetrachloroethylene (Perchloroethylene)	EPA 8260	Volatile Organics	NELAP	2/4/2002
Tetrachloroethylene (Perchloroethylene)	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Tetrachloroethylene (Perchloroethylene)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Tetrachloroethylene (Perchloroethylene)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Tetrahydrofuran (THF)	CA-202.08/GC-MS	Volatile Organics	NELAP	11/7/2006
Tetryl (methyl-2,4,6-trinitrophenylnitramine)	EPA 8330	Extractable Organics	NELAP	7/30/2004
Thallium	EPA 6010	Metals	NELAP	2/4/2002
Thallium	EPA 6020	Metals	NELAP	2/4/2002
Thallium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Thionazin (Zinophos)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Tin	EPA 6010	Metals	NELAP	7/30/2004
Titanium	EPA 6010	Metals	NELAP	7/30/2004
Toluene	EPA 8260	Volatile Organics	NELAP	2/4/2002
Toluene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Toluene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Toluene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Total cyanide	EPA 9012	General Chemistry	NELAP	4/26/2002
Total nitrate-nitrite	EPA 9056	General Chemistry	NELAP	2/4/2002
Total organic carbon	EPA 9060	General Chemistry	NELAP	2/4/2002
Total Petroleum Hydrocarbons (TPH)	FL-PRO	Extractable Organics	NELAP	2/4/2002
Total Petroleum Hydrocarbons (TPH)	TX1005	Extractable Organics	NELAP	9/4/2007
Total phenolics	EPA 9065	General Chemistry	NELAP	2/4/2002
Toxaphene (Chlorinated camphene)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Toxaphene (Chlorinated camphene)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Toxaphene (Chlorinated camphene)	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Toxicity Characteristic Leaching Procedure	EPA 1311	General Chemistry	NELAP	2/4/2002
trans-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	2/4/2002
trans-1,2-Dichloroethylene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004

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Matrix: **Solid and Chemical Materials**

Analyte	Method/Tech	Category	Certification Type	Effective Date
trans-1,2-Dichloroethylene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
trans-1,2-Dichloroethylene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
trans-1,3-Dichloropropylene	EPA 8260	Volatile Organics	NELAP	2/4/2002
trans-1,3-Dichloropropylene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
trans-1,3-Dichloropropylene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
trans-1,3-Dichloropropylene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
trans-1,4-Dichloro-2-butene	EPA 8260	Volatile Organics	NELAP	4/26/2002
Trichloroethene (Trichloroethylene)	EPA 8260	Volatile Organics	NELAP	2/4/2002
Trichloroethene (Trichloroethylene)	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Trichloroethene (Trichloroethylene)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Trichloroethene (Trichloroethylene)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Trichlorofluoromethane	EPA 8260	Volatile Organics	NELAP	2/4/2002
Trichlorofluoromethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Trichlorofluoromethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Trichlorofluoromethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Vanadium	EPA 6010	Metals	NELAP	2/4/2002
Vanadium	EPA 6020	Metals	NELAP	2/4/2002
Vanadium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Vinyl acetate	EPA 8260	Volatile Organics	NELAP	2/4/2002
Vinyl chloride	EPA 8260	Volatile Organics	NELAP	2/4/2002
Vinyl chloride	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Vinyl chloride	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Vinyl chloride	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Xylene (total)	EPA 8260	Volatile Organics	NELAP	2/4/2002
Xylene (total)	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Zinc	EPA 6010	Metals	NELAP	2/4/2002
Zinc	EPA 6020	Metals	NELAP	2/4/2002

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Matrix: **Solid and Chemical Materials**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Zinc	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006

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E87604
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Matrix: **Biological Tissue**

Analyte	Method/Tech	Category	Certification Type	Effective Date
4,4'-DDD	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
4,4'-DDE	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
4,4'-DDT	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
alpha-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1016 (PCB-1016)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1221 (PCB-1221)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1232 (PCB-1232)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1242 (PCB-1242)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1248 (PCB-1248)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1254 (PCB-1254)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1260 (PCB-1260)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
beta-BHC (beta-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
delta-BHC	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Dieldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endosulfan I	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endosulfan II	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endosulfan sulfate	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endrin aldehyde	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endrin ketone	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
gamma-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Heptachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Heptachlor epoxide	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Mercury	EPA 7470	General Chemistry	NELAP	7/30/2004
Mercury	EPA 7471	General Chemistry	NELAP	7/30/2004
Methoxychlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Toxaphene (Chlorinated camphene)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004

Laboratory Quality Manual

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In Accordance with:

Chapter 64E-1 Florida Administrative Code (FAC)
Certification of Environmental Testing Laboratories
And with the consensus standards adopted at the National Environmental Laboratory Accreditation Conference (NELAC)

This manual covers the following mobile units of KB Labs, Inc:

KB-1, KB-2, and KB-3

Effective Date: September 2008

Laboratory Quality Manual

Prepared by:

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CONCURRENCES:

KB Labs, Inc. Laboratory Director:
Signature: _____ Date: _____
Bradley A. Weichert

KB Labs, Inc. Quality Assurance Officer:
Signature: _____ Date: _____
Michael G. Winslow

EFFECTIVE DATE: September 2008

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1.0 STATEMENT OF POLICY AND OBJECTIVES

The policy of the management of KB Labs, Inc., is to implement a quality assurance program which is in compliance with the provisions and standards set forth in Chapter 64E-1 Florida Administrative Code (FAC), Certification of Environmental Testing Laboratories, which have been determined to be equivalent to the National Environmental Laboratory Accreditation Conference (NELAC) standards; and to assure that all certified environmental analyses are performed in accordance with the provisions and standards in Chapter 64E-1(FAC). The purpose is to ensure that all environmental data generated by KB Labs are scientifically valid, definable, and of known and acceptable precision and accuracy.

The management of KB Labs is committed to providing its clients services that conform to established quality requirements, including those associated with schedules and budgets, and assuring that all personnel strive to perform their job functions correctly without compromise of quality or obligations to clients.

1.1 SCOPE OF SERVICES

KB Labs provides a number of on-site analytical services using mobile laboratory facilities. KB Labs performs chemical analyses only. The primary focus is the determination of

- Volatile organic compounds (VOCs) by gas chromatography/mass spectrometry (GC/MS), providing full confirmation data on-site.

2.0 STAFF ORGANIZATION AND RESPONSIBILITIES

Figure 2-1 shows the organization and line of authority of KB Labs personnel.

2.1 DESCRIPTION OF JOB RESPONSIBILITIES

The job descriptions of key staff are described below.

- **President** - responsible for all contractual obligations of the proposed work and directs corporate efforts as necessary to achieve the objectives of schedule, cost, and technical performance. The President is also responsible for the review and administration of all contract changes, and for the direct communication and liaison with the client. The President can also act as a project manager.

- **Director of Operations** - responsibilities include the preparation of work plans and schedules, the allocation of manpower and material resources, and the direct communication with field team operations. The Director of Operations can also act as project manager.

- **Quality Assurance (QA) Officer** - provides monitoring and periodic internal auditing of the quality control (QC) procedures of the field chemists, ensures that established QC procedures are being followed, that adequate documentation is provided, and that all QC problems are handled in an expeditious manner. The QA officer is also responsible for the formatting and quality control of all documents and for the compiling, updating and submitting of the forms, SOPs, and the Laboratory Quality Manual.

- **Laboratory (Technical) Director -** responsible for the overall technical operations of all mobile laboratory units. The Laboratory Director is responsible for certifying that the field chemists with the necessary educational and technical training perform the analytical tests and maintain the overall operation of each mobile unit in accordance with the policies and procedures documented in the Laboratory Quality Manual.

- **Field Chemists** - responsible for performing quality analytical work in accordance with published standard procedures. Field chemists serve primarily as chemical analysts, but may also function as project managers, field team leaders, sample custodians, couriers, or other capacities on a project-specific basis. Field chemists are generally assigned the responsibility of operating and maintaining a single field mobile unit.

- **Health and Safety Officer** - responsible for the oversight of the laboratory health and safety program and maintenance of the Health and Safety Manual.

2.2 PERSONNEL EXPERIENCE AND TRAINING

Documented evidence for the following will be maintained on an ongoing basis for each member of the organization in designated personnel files kept in the administrative office of KB Labs. Copies of these files will also be maintained in each mobile lab facility. This evidence will include resumes, training records, demonstrations of capability, results of performance evaluation samples, etc.

- All personnel shall have sufficient education, training, experience and technical knowledge to adequately meet the requirements and responsibilities of their designated functions in the organization and they must comply with the specific quality requirements of their function.

- Technical personnel must be able to demonstrate a specific knowledge and skill in the performance of their technical tasks, as well as a general knowledge of analytical methods, laboratory operations, QA/QC procedures, and records maintenance.

- All technical personnel must have read and understood the Laboratory Quality Manual.

- All technical personnel must have read and understood all SOPs that address functions for which they are responsible.

- Field chemists must demonstrate on an annual basis proficiency in the test methods for which they perform. This proficiency requirement can be met with successful performance of a demonstration of capability, a blind PE sample, or at least four consecutive laboratory control samples.

Refer to KB Labs' Standard Operating Procedure (SOP) No. 027, *New Analyst Training.*

2.3 ETHICS TRAINING

All employees will receive instruction in the basic standards of ethical conduct that are expected of them while employed by KB Labs. This training will be conducted at the beginning of their employment and on an annual basis thereafter. The training will be conducted by the Lab Director or the Quality Assurance Officer and will include matters relating to data falsification or manipulation, client confidentiality, and professional conduct.

- An employee who falsifies or improperly manipulates data will be subject to termination of employment and/or possible legal action.

- All data generated, collected, or obtained from a third party subcontractor by KB Labs about KB Labs clients will be treated as confidential. No information or analytical data will be provided to a third party without the permission of the client.

- Employees should use “common sense” in complying with acceptable business practices and at a minimum adhere to the following:

    - Accept or give no gifts that where it could be inferred that business favors might be returned or expected by KB Labs.
    - Do not use information gained as a KB Labs employee for personal gain.
    - Make no promises that conflict with the employee’s responsibilities to KB Labs.
    - Report any violations of company policies.
    - Comply with federal, state, and local laws and regulations governing personal and business conduct.

- Employees must read and understand KB Labs’ SOP No. 029*, Ethics and Individual Responsibility Training*. A signed and dated copy of this document will be placed in the employee’s training file.

2.4 APPROVED SIGNATORIES

Table 2-1 below lists the approved title, current responsible party, and corresponding signature for laboratory document types.

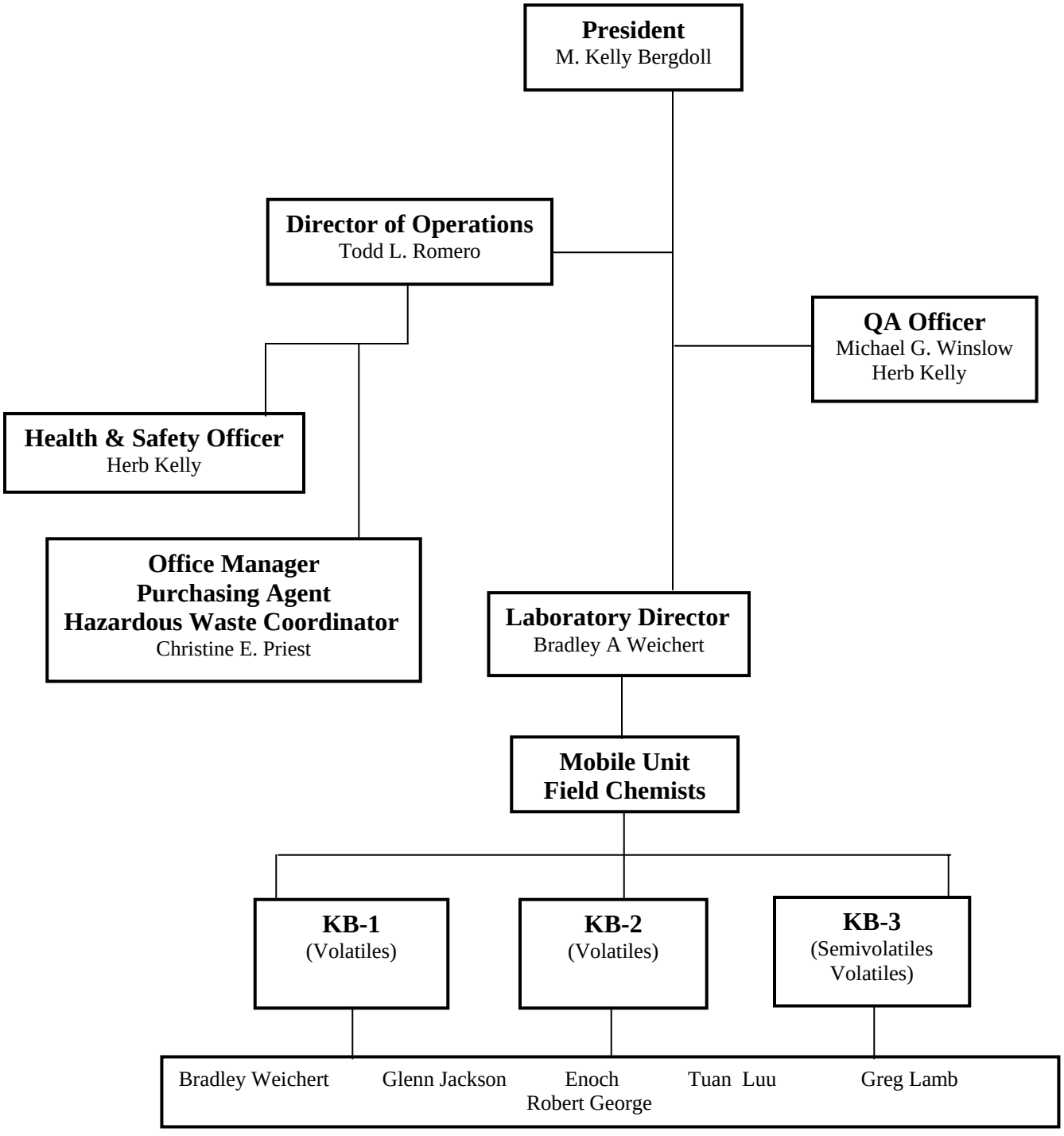


FIGURE 2-1: KB Labs Organization Chart

Table 2-1: Approved Signatories

Document Type	Title	Name	Signature	Initials
Laboratory Quality Manual	QA Officer	Michael G. Winslow		
	Lab Director	Bradley A. Weichert		
Bid, Proposals	President	M. Kelly Bergdoll		
	Director of Operations	Todd L. Romero		
Contracts	President	M. Kelly Bergdoll		
Reports to Clients	Director of Operations	Todd L. Romero		
	President	M. Kelly Bergdoll		
QA Reports	QA Officer	Michael G. Winslow		
	QA Assistant	Herb Kelly		
Preliminary Field Reports, Lab Notebooks, Logbooks, Data Sheets	Field Chemists	Bradley A. Weichert		
		Greg G. Lamb		
		Glenn Jackson		
		Enoch		
		Tuan Luu		
		Robert George		
Purchases	Office Manager	Christine Priest		

3.0 FACILITIES AND EQUIPMENT

KB Labs currently operates a total of three (3) mobile laboratories – two (2) dedicated to volatiles analysis, one (1) to semi-volatiles analysis and volatiles analysis. Each laboratory is designed to operate with a maximum of two field chemists, although generally for most projects only one chemist is required to operate a mobile laboratory facility. The mobile units are Izuzu single axle box trucks. Appendix A shows the floor plans for each of the mobile units with the arrangement of major analytical instrumentation and support equipment.

Tables 3-1 to 3-3 below list the major analytical instrumentation that is located in each of the mobile laboratories. Each mobile unit maintains the appropriate manuals supplied by the manufacturer for operation and maintenance of the instrumentation.

Each mobile unit operates as a stand alone laboratory and is NELAC certified as such.

Table 3-1 Major Instrumentation, KB-1

Item(s)	Model(s)	Serial Nos.	Year Purchased
Hewlett-Packard (HP) Gas Chromatograph/Mass Spectrometer/Data System	GC 5890A / MSD 5971A*/ Chem Station	3235A46501 (GC) 3188A02953 (MSD)	1998 1998
Hewlett-Packard Gas Chromatograph/Flame Ionization Detector/Integrator	GC 5890A / FID 19231 / Integrator 3396	2643A09969 (GC)	1998
Tekmar Purge & Trap Concentrator / 16 Position Autosampler	LSC 2000/ ALS 2016	90288012 (LSC) 90277001 (ALS)	1998 1998

* upgraded to 5972 in 2002

Table 3-2 Major Instrumentation, KB-2

Item(s)	Model(s)	Serial Nos.	Year Purchased
Hewlett – Packard (HP) Gas Chromatograph/Mass Spectrometer/Data System	GC 6890A / MSD 5973A / Chem Station	US00041726 (GC) US92511963(MSD)	2006
Hewlett-Packard (HP) Gas Chromatograph/Flame Ionization Detector /Integrator	GC 5890A / FID 19231 / Integrator 3396	2541A06416 (GC)	1999
Tekmar Purge & Trap Concentrator / Varian Autosampler	LSC 3000 / Arcon	94271006 (Tekmar) 90178025 (Varian)	2006

Table 3-3 Major Instrumentation, KB-3

Item(s)	Model(s)	Serial Nos.	Year Purchased
Hewlett-Parkard (HP) Gas Chromatograph/Electron Capture & Flame Ionization Detectors/Integrator/Data System	GC 5890 / Integrator 3396 /Turbo-chrom 4.0	2750A1644470	1999
Hewlett – Packard (HP) Gas Chromatograph/Mass Spectrometer/Data System	GC 5890A / MSD 5971A / Chem Station	2643A09843 (GC) 3306A04459(MSD)	1999
Tekmar Purge & Trap Concentrator / Autosampler	LSC 2000 / ALS 2016	90248015 (LSC) 90178025 (ALS)	1999
Applied Separations Pressurized Solvent Extractor	PSE 10502	032000401	2001

4.0 TEST METHODS AND STANDARD OPERATING PROCEDURES

Analytical reference methods and sample preparation reference methods currently performed by KB Labs, Inc. are listed in Table 4. The table also indicates the allocation of test methods among the different mobile units.

If additional, alternative, or modified procedures are ever proposed, a complete description of the method with data from an initial demonstration of proficiency will be provided to DOH for approval.

Each mobile unit maintains a copy of the most recent revision of the EPA SW846 reference method available for the tests performed in the mobile unit. In addition, a copy of the KB Labs analytical method SOP is attached to the published reference method. These SOPs document specific steps, procedural changes, and operating conditions actually utilized by KB Labs field chemists.

A comprehensive Laboratory Methods Manual is also maintained in the KB Labs administrative office that contains the latest revision of the SW846 methods used by KB Labs as well as copies of KB Labs analytical method SOPs.

4.1 ADDITIONAL SOPs

In addition to analytical method SOPs, KB Labs maintains copies of the following SOPs in both the mobile lab units and the administrative office:

SOP No.	Title
SOP001	Mobile Lab Power-Up
SOP002	Storage and Management of Gas Cylinders
SOP003	Final Report Preparation, Review, and Delivery
SOP004	Data Review and Validation
SOP005	Supply Requisition
SOP006	New Work Assignments
SOP007	Sample Receipt and Acceptance
SOP008	Filing and Archiving Project Records
SOP009	Significant Figures and Rounding Off
SOP010	Waste Disposal
SOP011	Temperature Monitoring

SOP No.	Title
SOP012	Storage of Standards
SOP013	Sample Storage
SOP014	Maintenance of Control Data
SOP015	Document Control
SOP016	Handling Complaints
SOP017	Corrective Actions
SOP018	Quality Control
SOP019	Labeling and Tracking of Standards
SOP020	Audits
SOP021	Sample Identification and Tracking
SOP022	Proficiency Test Samples
SOP023	Analytical Run Sequence
SOP024	Detection Limits
SOP025	Sample Containers, Preservation, and Holding Times
SOP026	Subsampling
SOP027	New Analyst Training
SOP028	Demonstration of Capability
SOP029	Ethics Training
SOP030	Contingency Plans for Changes in Ownership
SOP031	Protecting Confidentiality, Proprietary Rights, and National Security
SOP032	Departures from Documented Policies and Procedures
SOP033	Downtime Events
SOP034	Management Quality System Review
SOP035	Calculations
SOP036	Calculating and Reporting Soil Data
SOP037	Sample Receipt, Storage, and Disposal for Off-site or Fixed-base Analysis
SOP038	Subcontractor Pre-qualification Policy
SOP039	Weighing Soil Samples

SOP No.	Title
SOP040	Corrections to Entries
SOP041	Matrix Identification of Laboratory Control Samples
SOP042	Determining Measurement Uncertainty
SOP043	Manual Integration
SOP044	Calibration of Volumetric Dispensing Devices
SOP045	Limit of Detection

Table 4-1 Analytical Methods Performed by KB Labs, Inc.

Parameter	**Matrix**	**Sample Preparation Reference Method**	**Analytical Reference Method**	**KB Labs SOP No.**
Volatile organics (KB-1, KB-2, KB-3)	Water Soil	5030B 5035	8260B 8260B	KBSOP01VOC

5.0 PROJECT OPERATIONS

Figure 5-1 shows a flowchart of KB Labs overall project operations and primary task responsibilities from field trip preparation to data report delivery.

5.1 PROCEDURES FOR NEW WORK ASSIGNMENTS

Before accepting a new work assignment, the Director of Operations reviews the Request for Proposal (RFP) and/ or verbally questions the potential client to ascertain the following elements to determine if KB Labs, Inc. can successfully complete the work assignment:

- Analytical compounds to be analyzed and by what analytical method.
- Determine sample matrix (i.e., ground water, soils, etc.)
- Approximate number of samples to be analyzed.
- Time duration and site location of project.
- Requested reporting limits.
- Point-of-Contact (POC) information
- Final reporting requirements (both electronic and hardcopy).

The following elements are reviewed by the Director of Operations as the basis for mobile laboratory and Field Chemist assignment to new project:

- Analytical compounds to be analyzed and analytical method.
- Training and analytical experience of Field Chemist.
- Availability of laboratory and Field Chemist for scheduled project dates.
- Past chemist experience at site and/or previous interaction with client.

Preparation of the laboratory and Field Chemist for new work assignment involves the following:

- Field Chemist is issued completed Work Order Form (See Appendix B) from the Director of Operations (Project Manager).
- Required standards, gases, and laboratory supplies are issued (if necessary) to the assigned laboratory by the Director of Operations.
- Routine maintenance is performed on the laboratory and analytical instruments prior to departure by the Field Chemist.

See also KB Labs' SOP No. 006, *New Work Assignments*.

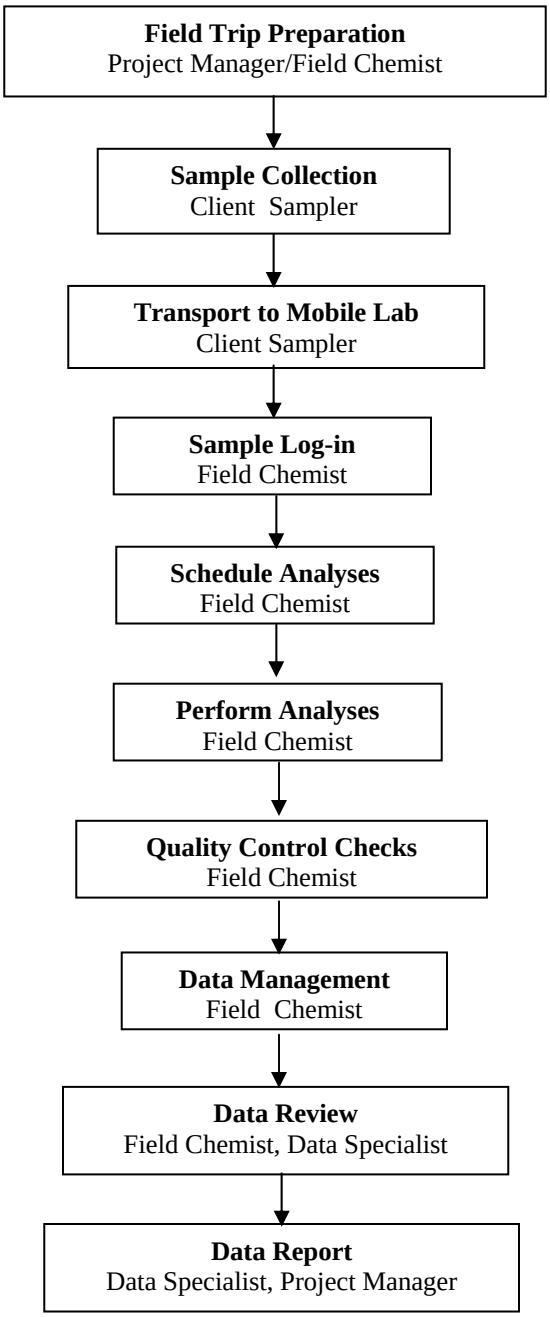


FIGURE 5-1: Flowchart of KB Labs Project Operations

6.0 SAMPLE TRACKING AND HANDLING`

6.1 SAMPLING

KB Labs provides no sampling services. However, KB Labs will supply sample containers. All sample containers are purchased pre-cleaned (and with preservatives if required) from certified commercial suppliers. Sample containers are not cleaned for reuse.

Table 6-1 lists containers, preservation methods, and holding times for volatile and semi-volatile sample types.

6.2 SAMPLE CUSTODY

Sample custody is an essential part of field and laboratory operations and is defined as follows:

Sample custody – the sampler or transferee is in physical possession of the sample or was in physical possession of sample and sample was then placed in a secure area to prevent tampering. Where data may be needed for potential litigation, strict **chain-of-custody** procedures must be used.

6.3 CHAIN-OF-CUSTODY

A Chain-of-Custody Record (see Appendix B) is initiated at the time sample containers are dispatched to the field sampling team by the field chemist. A Chain-of-Custody Record accompanies sample containers to the field. This document is used by the field team to record sample identification, sample description, date, time, and location of collection, analyses required, and condition of sample. All requested information on this form must be completed where appropriate. This document must be signed by a member of the field team. All errors are deleted with one line through error and initialed and dated.

However, in order to meet the NELAC requirement that each sample have a **unique sample identification number** to facilitate and insure accurate sample tracking if needed at a later date, and because clients often repeat sample identification schemes from site to site, KB Labs will identify samples by combining the mobile lab identification, sampling date, and client field ID – e.g. for field sample SB-1, analyzed in mobile unit KB-1 and received from the client on January 1, 2006, its unique identification will be KB1*010106*SB-1. Refer to KB Labs SOP No. 021, *Unique Sample Identification*.

6.4 SAMPLE RECEIPT

The field chemist is designated as the sample custodian. This person receives samples from a member of the field sampling team and checks for the following:

- appropriate sample containers.

- adequate sample volume or mass
- signs of leaking, broken, or contaminated sample containers.
- whether headspace is present in the sample container (VOCs).
- proper preservation as specified in Table 6.
- complete documentation and identification of samples, and signature of field sampler on the Chain-of-Custody Record
- proper sample labeling

If any of the previous conditions are not properly met, the improper conditions for each sample will be noted on the Chain-of-Custody Record and will be reported to the field team leader. The field team leader will then make the decision on whether to reject the sample(s). After verifying the status of the samples, the sample custodian (field chemist) signs the sample Chain-of-Custody Record. The original copy is kept with the project file, a copy is placed in the Sample Receipt logbook, and another copy is sent to the client.

Refer to KB Labs' SOP No. 007, *Sample Receipt and Acceptance*.

6.5 SAMPLE STORAGE

All samples are stored in wet ice in coolers kept at $\leq$ 6 °C. Prior to sample preparation or analysis, samples are retrieved from the cooler by the field chemist and allowed to come to room temperature before analysis. The samples are returned to the cooler upon completion of sample preparation or analysis. (VOC samples are not returned to the cooler after aliquots are removed for processing. However, duplicate samples do remain in the cooler until disposal.)

The field chemist has the ultimate responsibility of ensuring analytical holding times are met for each project. All samples are secure in the laboratory with access only available to members of the staff of KB Labs and designated sample custodians. All samples are stored well away from standards. All samples for VOC analyses are stored on ice in a separate cooler at $\leq$ 6 °C. No other samples, reagents, extracts or standards will be stored in this cooler.

Refer to KB Labs' SOP No. 013, *Sample Storage*.

6.7 SAMPLE DISPOSAL

Unused samples should be returned in their containers to the client field samplers at the conclusion of the job, before the mobile lab leaves the project site . Purged and extracted samples and solvent extracts will be stored in clearly labeled approved containers and disposed of in accordance with DEP approved procedures.

Refer to KB Labs' SOP No. 013, *Waste Disposal*.

Table 6-1: Sample Containers, Preservation Methods, and Holding Times*

Parameter	Matrix	Container	Preservative**	Holding *** Time (days)
Volatiles	Water	Glass vial, screw cap with Teflon™-lined septum, 2 x 40 mL	Cool, ≤ 6 °C PH < 2	14
	Soil	Glass jar, Teflon™-lined screw cap, 4 oz.	Cool, ≤ 6 °C	14
Semivolatiles	Soil	Glass jar, Teflon™-lined screw cap, 4 oz.	Cool, ≤ 6 °C	14 extraction 40 analysis

* From 40 CFR Part 136 Table II and Chapter 62-160 F.A.C.

** Sample preservation should be performed immediately after sample collection.

*** Samples are analyzed on-site, generally within 24 hours of collection.

7.0 CALIBRATION AND TRACEABILITY OF MEASUREMENTS

Because measuring operations employing analytical instruments and other support test equipment have an effect on the accuracy or validity of tests, each mobile unit must perform calibration and verification procedures before equipment is put into service and on continuing basis.

7.1 INSTRUMENT CALIBRATION

Instrument calibration procedures establish the relationship between a calibration standard of known concentration and the measurement of the standard concentration by an instrument or analytical procedure. At a minimum, calibration is required (1) when an instrument is first started up; (2) daily, prior to the analysis of a batch of samples, (3) when the instrument has been subject to major maintenance, or (4) when the instrument fails the calibration quality control checks.

Initial calibration is performed when the instrument is started up or when the instrument response has drifted out of calibration in order to demonstrate that the instrument is capable of acceptable performance at the beginning of the analytical run and is producing a linear calibration. Initial calibration is usually performed with five standards that cover the analytical working range of the method. The standard concentrations will be adjusted to take into account the instrument and method, the upper and lower limits of linearity, and the instrumental detection limit.

Continuing calibration is performed at the beginning and/or every 12 hours in order to verify initial calibration. The continuing calibration standard (CCS) is generally a mid-level standard from the initial calibration but should be varied within the calibration range on a regular basis.

(Sample responses are quantitated from the initial calibration and not from continuing calibration.)

In all cases, if the method calibration requirements are more stringent than those listed in this document, then the method calibration requirements will be followed. In all cases, when an instrument is calibrated for analysis it will be recorded in an instrument logbook with date, initials of analyst, analyte(s), and all appropriate instrument settings. It will also be recorded on the analytical bench sheet or computer printout how the instrument was calibrated.

7.2 PREPARATION OF INSTRUMENT CALIBRATION STANDARDS

Stock solutions used to prepare calibration standards, surrogate and matrix spike solutions, and internal standard solutions are purchased from commercial suppliers (see Table 7-1 below).

For stock standards purchased directly through a supplier, the initials of the receiver and date of receipt are written in ink on the original Certificate of Analysis. If the standard has an expiration

date, this date is circled in ink to ensure that the preparer does not use expired standards. As these expire, they are disposed. All information concerning these standards, including LOT#, supplier of standard, concentration of standard, purity of standard, and method of determination of purity are on the original container and the Certificate of Analysis which accompanies the standard. Further information concerning the purchase of the standards is in a purchase order logbook with date of purchase, purchaser of standard, supplier of chemical and date of receipt of standard. All other information concerning these standards can be obtained from the supplier of the standard as needed. The original copy of the Certificate of Analysis for stock standards purchased in kept on file with the Quality Assurance Officer.

Working standards are prepared directly from the stock standard. If required, all dilutions are prepared in Class-A volumetric glassware. All documentation tracing the working standards to stock standards and chemicals will be kept in a standards notebook next to each instrument and will include the analyte(s), initials of analyst, date of preparation, concentration levels of standards, how standards were prepared, and stock standard used to prepare working standards and intermediate standards if applicable.

Working standard solutions will be prepared by sequential dilution of a single stock standard to bracket the analytical working range of the method. Working standard solutions may be either composite standards of more than one analyte or single-analyte solutions. The standard concentrations will be adjusted to take into account the instrument and method, upper and lower limits of linearity, and the instrumental detection limit. At least three (3) standard concentrations covering the working range and a blank will be prepared and analyzed. The working standards and the blank will be analyzed at the beginning of the analytical run (initial calibration) and at least one mid-level standard will be reanalyzed at least every 12 hours and at the end of the run to check for constant instrument response (continuing calibration verification).

7.3 STANDARD CURVE CALIBRATION

The working curve will be produced by plotting the standard response for each standard versus the concentration of each standard from the initial calibration run or average response factors, if method criteria are met. Specific quality control acceptance criteria for working curves or for continuing calibration standards are listed in the methods and will be followed.

7.4 INTERNAL STANDARDS (GC/MS)

Internal standards are added to all samples and standards that are analyzed for GC/MS analysis. Quantitation cannot be performed without internal standards. Method appropriate internal standards are listed in the analytical reference method. The working concentration of the internal standards will be prepared according to the method.

7.5 INSTRUMENT TUNING (GC/MS)

Daily instrument tuning will be performed to ensure that the instrument is calibrated and in proper working condition. Bromofluorobenzene (BFB) will be used as the tuning compound for volatile analysis and the mass intensity specifications will be followed according to each method. The working concentration for BFB will be prepared according to the method.

7.6 STORAGE OF CALIBRATION AND REFERENCE STANDARDS

All standards are stored in refrigerators located in each mobile laboratory facility. Standards will be stored separately from samples. VOCs will be stored in a freezer at $-10\ ^{o}$C and SVOCs and metals in a refrigerator at ≤ 6 °C.

Refer to KB Labs' SOP No. 013, *Storage of Standards*.

7.7 MONITORING OF REFRIGERATORS AND FREEZER

Temperatures for refrigerators and freezers are measured on a daily basis and recorded in a Daily Temperature Record . If the measured temperature for this equipment is out of control, it will be noted in the logbook, the necessary adjustment will be made to correct the temperature and it will be monitored until temperature is in control and constant. All laboratory thermometers are calibrated annually against a NIST certified thermometer. If any thermometer is more than 1^{o}C different from the NIST thermometer, it is replaced.

Table 7-1: Standard Sources and Preparation

Type	Standard Source	Preparation From Source	Lab Stock Storage	Preparation Frequency
Calibration compounds	Purchased from supplier	Working solutions are made directly from source stock	Refrigerator @ ≤ 6 °C (SVOCs) Freezer @ -10 °C (VOCs)	Weekly for gases, or monthly
GC/MS Internal standards (VOCs)	Purchased from supplier	Working solutions are made directly from source stock	Freezer @ -10 °C	Semiannually
Matrix spike and surrogate compounds	Purchased from supplier	Working solutions are made directly from source stock	Refrigerator @ ≤ 6 °C (SVOCs) Freezer @ -10 °C (VOCs)	Semiannually
GC/MS Tuning Compound Bromofluoro-benzene	Purchased from supplier	Working solutions are made directly from source stock	Freezer @ -10 °C	Annually

8.0 SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA ACCURACY AND PRECISION

Data **accuracy** will be assessed for each measurement system and each sample lot using a known reference sample and/or a sample spiked at a known level. The recovery of the sample will then be compared to the method accuracy acceptance criteria established by the laboratory.

Data **precision** will be assessed similarly using replicate analyses. Data precision will be compared to the method precision acceptance criteria established by the laboratory.

If the accuracy or precision results do not fall within the established control limits for method performance, then the results reported for all samples processed as part of the same set must be labeled as suspect, and the samples may need to be repeated. The project QA officer and project manager will be notified and the necessary corrective action implemented.

In all cases, if the EPA method specific QC requirements (if established) are more stringent than those established by KB Labs, then the method QC requirements should be followed.

8.1 LABORATORY QUALITY CONTROL CHECKS

Types of QC samples used include method blanks, matrix spikes, matrix spike duplicates, laboratory control or reference spikes, surrogates, and blind performance evaluation samples.

The following minimum QC checks will apply to all analyses:

Method blank – Daily analysis of laboratory reagent water or standard soil samples is performed in order to monitor the cleanliness of the analytical system. Method blank analysis (VOCs only) is performed before analyzing samples, after high level sample analysis, and at least every 12 hours. For SVOCs there should be at least one method blank for every batch of 20 or less samples extracted.

Matrix spike/matrix spike duplicates (MS/MSD) – A known amount of each target compound is added to duplicate aliquots of a selected field samples in order to monitor the performance (precision and accuracy) of the target analytes in an actual matrix. An MS/MSD is analyzed at a frequency of one pair every 20 samples of a matrix type (soil or water).

Reference standard/laboratory control spike (REF/LCS) – A REF/LCS is analyzed after the initial calibration to check the validity of the calibration standards. The REF/LCS is prepared from a different source stock standard than are the calibration standards. An REF/LCS is analyzed at a frequency of one for every preparation batch of 20 or less samples of a matrix type.

Surrogate standards – The surrogate standard solution is added to all samples and standards that are analyzed. The surrogate compounds evaluate the performance of the analytical system and to help determine the potential for sample matrix effects.

QC tables are maintained for duplicate spikes, and reference samples. Separate tables are maintained for each analytical method. Warning and control limits are established by standard deviation techniques.

For duplicate samples, the relative standard difference (RPD) = $\frac{|X_1 - X_2|}{(X_1 + X_2 / 2)} \times 100$

is utilized as the test statistic precision.

For spike data, the test statistic for accuracy is the percent recovery of the spike defined as follows:

$$\% \text{ Recovery} = \frac{\text{conc. spiked sample} - \text{conc. unspiked sample}}{\text{conc. of spike actually added}} \times 100$$

The test statistic for reference samples is the actual measured concentration. For both of these test statistics the mean and standard deviation are determined utilizing a number of data points. The warning and control limits are established as ± 2 and ± 3 standard deviation units from the calculated mean values, respectively. These limits are updated on a continuing basis as new QA data is entered. They are based on the most recent 50 data points for a given analyte and matrix. If a sufficient number of QA data points is not available for a given analyte and matrix, then the QA targets will be based upon published QA targets until sufficient data points have been generated.

Refer to KB Labs SOP No. 018, *Quality Control*.

8.2 METHOD PERFORMANCE

Method performance is established by determining the **Method Detection Limits (MDLs)** in the matrix of interest. The MDL is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDL that is achieved for a given analyte will vary depending on instrument sensitivity and matrix effects.

The MDL for both waters and soils is experimentally determined by KB Labs using procedures described in 40 CFR, Part 136, Appendix B and as per 91-04. Seven replicate samples of each matrix (standard laboratory reagent water or soil) are spiked with a known concentration of each analyte of interest. The concentrations for each analyte are then experimentally determined using the procedures described above for this method. The standard deviation of the found concentrations for the seven replicates is then calculated. The MDL for each analyte is then determined by multiplying the standard deviation by 3.14.

Refer to KB Labs' SOP No. 024, *Detection Limits*.

Laboratory control limits are established by KB Labs for both waters and soils. The laboratory control limits are calculated by determining the average percent recovery and standard deviation measured for each analyte when determining its MDL. The upper and lower control limits are calculated as the average percent recovery plus or minus 3 times the standard deviation.

All MDLs will be verified or updated on an annual basis. Copies of the MDL studies for each method will be kept in the appropriate mobile units and in the mobile unit files maintained in the administrative office.

Refer to KB Labs' SOP No. 014, *Maintenance of Control Data*.

8.3 DEMONSTRATION OF ANALYTICAL CAPABILITY

Each analyst, prior to using any test method on samples, must perform a demonstration of capability for each method. This requirement will also hold for any time a new instrument is introduced into the laboratory.

The demonstration of capability will consist the same procedures followed for determining the MDLs as described in Section 8.2 above. A minimum of four (4) replicate samples (instead of 7) are required.

All demonstrations of capability will be documented on the Demonstration of Capability form shown in Appendix B. These completed and signed forms will be maintained in each analyst's training and experience file.

Refer to KB Labs' SOP No. 028, *Demonstration of Capability*.

9.0 DATA REDUCTION, VALIDATION, AND REPORTING

9.1 DATA REDUCTION

Data reduction and transfer are essential functions in summarizing information to support conclusions. It is essential that these processes are performed accurately and that accepted statistical techniques are used. Field chemists are responsible for calculating final data and QC data from raw data recorded on laboratory bench sheets, chart recordings, and computer printouts. All calculations are in accordance with the approved methods cited earlier. Example calculations are included with summarized data to facilitate review. All computer printouts should be labeled with analyst name, analyte, date of analysis, project name, and all pertinent instrument settings. All data are compiled in a project file folder for delivery to the Data Specialist for review. To facilitate data review for each project, the following items should be in each project file folder in the order listed:

- Diskette with field preliminary data report in electronic spreadsheet format
- Work Order Form
- Field log sheets
- Chain-of-custody sheets
- A hardcopy of the field preliminary data report
- Field Chemist Comments (See Appendix B) – any pertinent comments such as departures from method, problems, etc.
- Spike recovery summaries
- Initial multilevel calibration summaries (if performed)
- Tune records (MS)
- Daily sequence summaries
- Instrument analysis chromatograms and quantitation reports in chronological order
- Screening chromatograms

9.2 DATA REVIEW AND VALIDATION

Data review and validation is conducted by the Data Specialist who is responsible to the designated project manager. All work performed by the Field Chemist is checked during the data review process. The signature of the reviewer and date of review are entered on the Data Review Checklist (see Appendix B) each project file folder. The responsibility of the Data Specialist is to ensure the following:

- Each project folder has the items listed in Section 9.1 above.
- All data are calculated correctly.
- All data are entered correctly in the field data report.
- All QC data are calculated correctly.
- All QC values are within the acceptance criteria.
- Check that chain-of-custody forms are properly completed and that sample identifications are in agreement with those in the project file and data report.

Upon completion of data review and validation, the Data Specialist will include a Data Folder Table of Contents Checklist (see Appendix B and a Data Review Checklist in the project data file .

Refer to KB Labs' SOP No. 004, *Data Review and Validation*.

9.3 DATA REPORTING

The Data Specialist generates the final report to the client and is responsible to the designated project manager. Assurance that reported data are correct is the responsibility of the project manager, the Data Specialist, and the Field Chemist. The final report to the client contains the following:

- A cover letter which references the project name, date, and location and summarizes the contents of the report and the qualifications of KB Labs, Inc
- A brief project narrative which provides general information about the project scope, analytical procedures, analytical results, and QC data
- A data narrative addressing discrepancies between the final data report and the preliminary field data report
- A table listing the analytical run sequence with surrogate recoveries
- A table listing matrix spike and control spike recoveries
- A final data report in spreadsheet format
- Copies of the chain-of-custody sheets

Appendix C gives an example of a standard KB Labs final report to client.

Refer to KB Labs' No. 003, *Final Report Preparation, Review, and Delivery*.

10.0 SYSTEM AND PERFORMANCE AUDITS

Systems and performance audits are used to assess and document the performance of field chemists. These audits form a basis for corrective action requirements and constitute a permanent record of the conformance of measurement systems to QA requirements. Refer to KB Labs' SOP No. 020, *Audits*.

10.1 INTERNAL SYSTEM AUDITS

The QA Officer will choose random sample numbers from different projects conducted during the year and trace the sample from receipt to final reporting, reviewing proper chain of custody, sample receipt procedures, proper method selection, data reduction, sample preparation, and analysis within holding times. At least one sample will be audited annual for each method and mobile unit. Completed Internal Audit Reports will be submitted to the Lab Director and to the President.

All laboratory QC information will be reviewed at least annually by the QA officer. This information, including replicates, spikes, and reference samples for all methods is printed and kept on file.

The QA officer checks all instrument and analytical logs to assure that all analytical work that has been done is properly documented, including date of analysis, initials of analyst, analyte, and all pertinent instrument settings.

10.2 EXTERNAL SYSTEM AUDITS

KB Labs is biannually inspected by FL DOH, and all recommendations are implemented. KB Labs is open to FL DOH for inspection at any time.

10.3 INTERNAL PERFORMANCE AUDITS

Internal performance audits consist of commercially produced QC check samples (Laboratory Control or Reference Samples) run with each analysis by the analyst. The found value is then compared to the true value. All QC check sample data is entered onto the analytical bench sheet including true value, found value, percentage recovery and, if applicable, 95 percent confidence interval. The QC check sample will be from a different stock source than the calibration standards.

10.4 EXTERNAL PERFORMANCE AUDITS

Biannual performance audits include the analysis and evaluation of **proficiency test samples**. KB Labs will participate in the U.S. EPA Water Pollution Laboratory Performance Evaluation study Program. Results of these analyses will be provided to DOH.

Refer to KB Labs' SOP No. 022, *Proficiency Test Samples*.

11.0 CORRECTIVE ACTION

Data acceptability is based on the quality assurance objectives for measurement data stated in Section 8.0.

If the acceptance criteria are not met for one or more QC checks, than the first person to take corrective action will be the analyst. The analyst will determine when the system was no longer in control and follow the corrective action.

If the system is still out of control, the Laboratory Director and QA officer will be informed of the specific discrepancies and decisions concerning the data will be made on a project specific basis.

The Laboratory Director or QA officer will be responsible for notifying laboratory personnel of the corrective action(s) be taken.

For external QC discrepancies (performance evaluation sample), the Laboratory Director and QA officer will determine the source of the discrepancy, plan a course of action to solve the problem and inform the appropriate laboratory personnel of the new procedure.

All DOH recommended corrective actions will be initiated as a result of system or performance audits, split samples or data validation review.

Refer to KB Labs SOP No. 17, *Corrective Actions*.

11.1 COMPLAINTS

Whenever a complaint is received by KB Labs from a client or other party about compliance with the NELAC Standard, project requirements, laboratory policies and procedures, or about the quality of the laboratory's test results, an internal audit (Sec. 10.1) will be immediately conducted by the Quality Assurance Officer, or in the case of specific project issues unrelated to data quality, the Director of Operations will investigate the problem. A record of the complaint and subsequent action will be maintained in the project file in the administrative office.

Refer to KB Labs SOP No. 16, *Handling Complaints*.

11.2 DEPARTURES FROM PROCEDURES AND SPECIFICATIONS

It is the policy of KB Labs that documented procedures and standard specifications will be followed on a routine basis for all projects. However, the following exceptions may arise:

- If a client requests a departure from a documented procedure or standard specification, it will be noted in detail on the report to the client and in the project file.
- If KB Labs cannot, because of unexpected field conditions or operational circumstances, follow a documented procedure or a standard specification, the Field Chemist will immediate notify the Director of Operations and/or the Quality Assurance Officer, who

will then notify the client. The departure will be documented in the final report to the client and in the project file.

Refer to KB Labs' SOP No. 32, *Departures from Documented Policies and Procedures*.

Table 11-1: Corrective Action

QC Activity	Acceptance Criteria	Recommended Corrective Action
Initial Calibration	Follow protocol stated in reference method.	Rerun calibration. If necessary, prepare fresh calibration standards.
Method Blank	$\leq$ 1/10 concentration in any sample associated with blank.	Re-prep and reanalyze new method blank and samples associated with contaminated blank. If necessary, perform appropriate instrument maintenance.
GC/MS Tuning	BFB ion abundance criteria must be met as set forth in Method 8260b	Perform mass calibration. Retune hardware. If necessary, clean source.
Continuing Calibration	Follow protocol stated in reference method.	Rerun continuing calibration standard. If necessary, rerun initial standard calibration.
Surrogates	Within established control limits	Reanalyze samples that have one or more surrogates out of control.
MS/MSD Spikes	Within established control limits	Reanalyze samples that have one or more spikes out of control.
LCS	Within established control limits	

12.0 RECORD KEEPING AND DOCUMENT CONTROL

12.1 STORAGE OF PROJECT DATA

All **project data files** containing the items listed in Section 9.1 above are stored chronologically in a file cabinet in KB Labs' administrative office. No project data files are stored in the mobile laboratory units. In order to help maintain the integrity of data and to protect the confidentiality and proprietary rights of all clients, these files are archived as needed into banker's boxes that are kept in a secure storage area of the administrative office. All archived data are stored at least 5 years in accordance with NELAC standards. An access log will be maintained for retrieving the archived files. Access is limited to employees of KB Labs only. No data will be released to parties other then the client without written permission from the client.

Data from the analytical instrument computers are archived onto backup disks at least every six months. These disks are stored in the administrative office in fire proofs boxes in a designated area. They are labeled with the project number and project name and stored sequentially by project number.

Final reports to the client (see Section 9.3) are stored in electronic computer files by client and project name. These are regularly backed up on disk and stored in the administrative office fire proof boxes in a designated area. A photocopy of the final report to the client is also contained in the project management file, which is described below in Section 12.2

Refer to KB Labs SOP No. 008, *Filling and Archiving Project Records*.

12.2 STORAGE OF ADMINISTRATIVE RECORDS

Project management files are maintained by the Director of Operations in a separate file cabinet in the administrative office. These files are arranged alphabetically by client and within the client file, alphabetically by project name. A project management file generally contains the following documentation:

- Proposal or bid
- Contract
- Work Order
- Final Report
- Correspondence
- Notes

Personnel files are maintained alphabetically by the QA Officer in a separate file cabinet in the administrative office and contain the following documentation:

- Resume
- Training Record
- Demonstrations of Capability

Mobile unit files are maintained by the QA Officer in the administrative office. These files contain the following documentation:

- SOPs for analytical methods performed in the mobile unit
- MDL study data
- Copies of all demonstration of capability certifications
- PE sample data

12.3 MOBILE UNIT RECORD KEEPING

Each of the mobile units will maintain limited documentation for operations conducted in the unit. This documentation will include the following, each of which will be maintained in separate, labeled folders stored in a file cabinet:

- SOPs for analytical methods performed in the mobile unit
- Other company SOPs
- Most recent MDL study data for each method performed in the unit
- Copies of all demonstration of capability certifications for unit personnel
- PE sample data for at least the last three rounds for each analytical method performed
- Personnel Resume(s)
- Personnel Training Record (s)

In addition, each mobile unit will maintain a copy of the most recent version of the Laboratory Quality Manual and Health & Safety Manual and will maintain logbooks for sample receipt, instrument run, instrument maintenance and repair, standards, and refrigerator temperature. Completed logbooks will be stored in the administrative office in a designated area.

12.4 DOCUMENT CONTROL

Official KB Labs documentation such as the Laboratory Quality Manual, Laboratory Health & Safety Manual, SOPs, standard forms, etc. are updated annually or whenever necessary. It is important that the most recent revision of each document is utilized by all personnel. In order to facilitate this, each document will have the document file name, date of production, and revision number clearly indicated in the header or footer.

It is the responsibility of the Quality Assurance Supervisor to assure that all official documents are updated as required and that the latest revision is in use by all personnel. Copies of old outdated documentation will be kept on file by the QA Officer. A master list of the latest revision of all documentation will be maintained and posted in the administrative office by the Quality Assurance Supervisor.

Refer to KB Labs SOP No. 015, *Document Control*.

13.0 PREVENTIVE MAINTENANCE

To minimize the occurrence and severity of instrument failure, a preventive maintenance program for laboratory instruments has been implemented. The preventive maintenance performed for major pieces of analytical equipment is listed below in Table 13-1.

13.1 DOCUMENTATION OF ROUTINE MAINTENANCE AND NON-ROUTINE REPAIRS

All repairs are documented in the instrument logbook, which includes the date of maintenance or repair and description of work. Further documentation is provided in the instrument file, which includes complete documentation of repair work completed.

In the event of any instrument failure KB Labs will proceed with the following actions:

1. Repair of instrument by staff
2. Repair of instrument by service representative
3. Return instrument to place of manufacture for repair
4. Acquire new instrumentation

In the event of excessive down time, KB labs will either acquire instrumentation to complete project work or subcontract project work to fulfill project requirement.

13.2 LABORATORY SET-UP ROUTINE

Each time a mobile lab unit is relocated, the following setup routine is following:

1. Verify connection to generator fuel supply and trailer ground strap.

2. After starting generator, check voltage output. Voltage should be 120 $\pm$ 10 volts.

3. Turn on climate control.

4. Turn on analytical instruments and allow heated zones to come to temperature. (After turning on GC/MS, pump down MSD (approximately 2 to 4 hours) to operating vacuum. Verified by the ion gauge controller.)

5. Check GC gas flow leaks settings . Check MS system for leaks if system will not pump down or if there is excessive noise in baseline.

6. Prepare reagent water blanks and analyze system blank. If blank passes, initial calibration can begin. (GC/MS must also pass tune check before sample analysis can begin.)

Table 13-1: Preventive Maintenance Procedures

Procedure	Frequency
Gas Chromatograph	
Change septa	When system develops leaks or as needed
Check carrier gas	Daily
Change carrier gas	When pressure falls below 100 psi
Cut edge of capillary column	When system performance declines
Change columns	When column performance declines or as needed
Change injector port liner	When dirty or as needed
Mass Spectrometer	
Backup system software	Monthly
Replace traps	Annually
Manufacturer's preventive maintenance	Annually
Clean source	When calibration compound criteria cannot be achieved or as needed
Keep instrument clean and dust-free	After each use
Purge and Trap	
Clean, bake, and purge spargers	Daily prior to use
Bake out trap	Daily prior to use
Replace trap	Quarterly or as needed
Replace fittings	Annually or as needed

Table 13-1: Preventive Maintenance Procedures (Cont'd)

	Procedure	Frequency
Support Equipment		
Ovens, Refrigerators	Monitor temperature, keep units clean	Daily
Hot Plates	Keep units clean	After each use
Generator	Check oil level	Before starting
	Check battery fluid	Monthly
	Change oil and filter	Every 1000 hours
	Change air filter	Monthly, more often if conditions are dusty
	Check voltage	Upon startup

APPENDIX A

Floor Plans

Figure 3-1: Floor Plan, KB-1

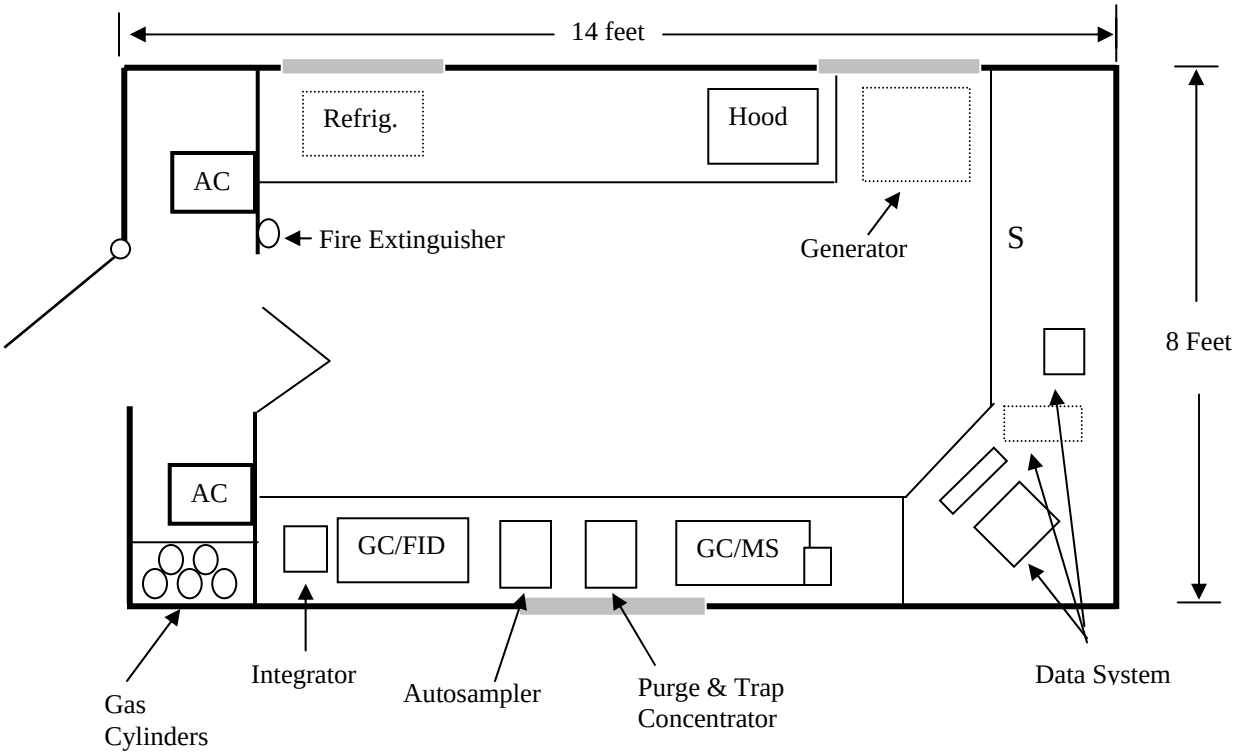


Figure 3-2: Floor Plan, KB-2

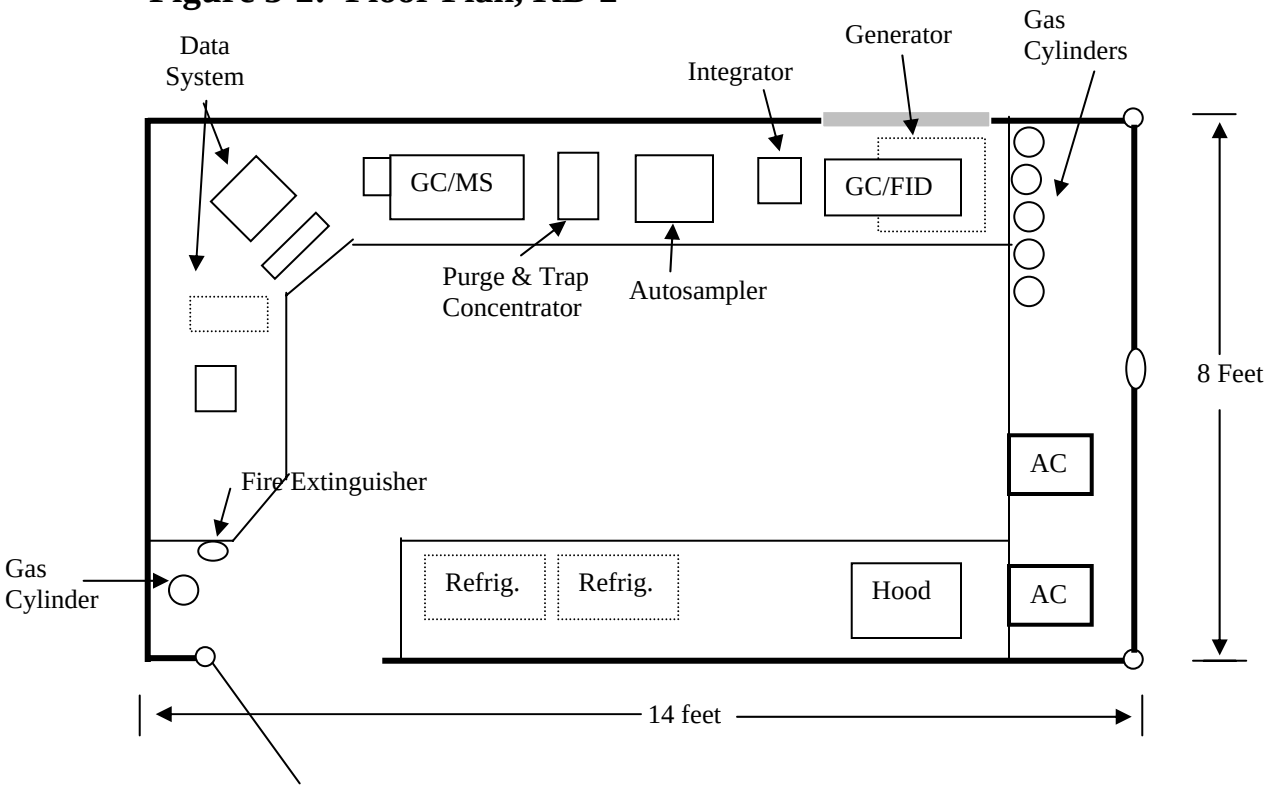
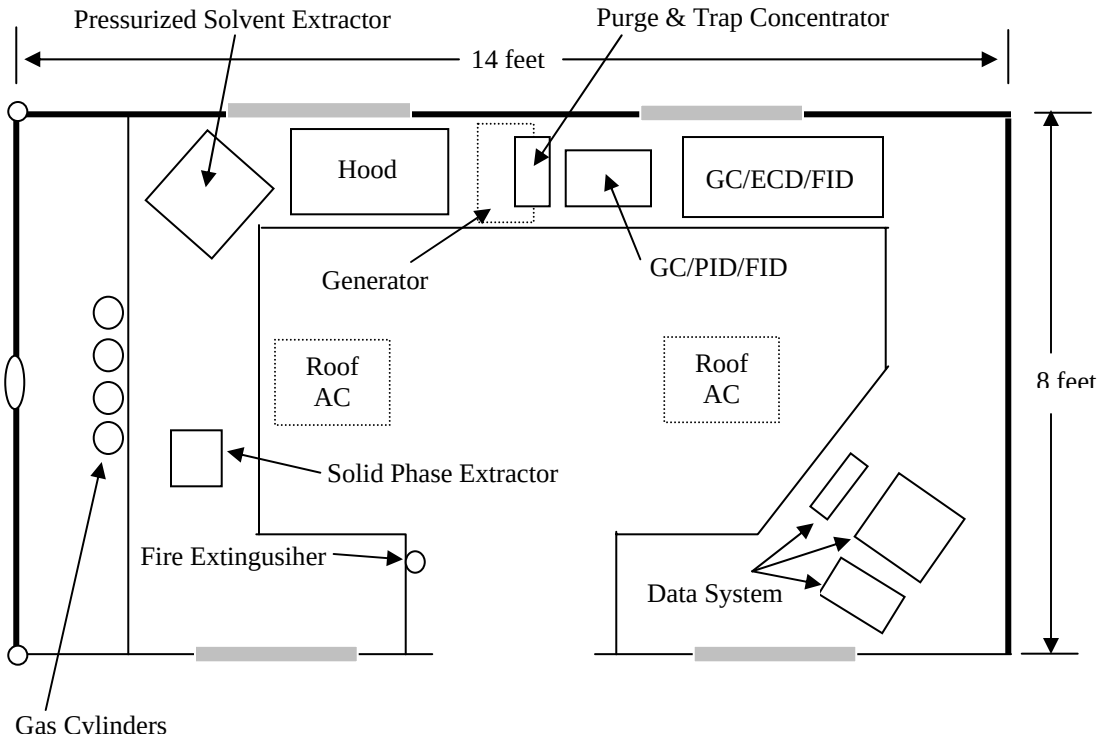


Figure 3-3: Floor Plan, KB-3



APPENDIX B

Standard Forms

Demonstration of Capability
Certification Statement

Date:	**Analyst Name:**	**Mobile Lab No:**
Matrix:	**Method Number:**	**SOP Number:**
Parameters:		

We, the undersigned, CERTIFY that:

1. The analyst identified above, using the cited test method, which is in use at this facility for the analysis of samples under the National Environmental Laboratory Accreditation Program, has met the Demonstration of Capability.

2. The test method was performed by the analyst identified on this certification.

3. A copy of the test method and the laboratory-specific SOPs are available for all personnel on-site.

4. The data associated with the demonstration of capability are true, accurate, complete and self-explanatory.

5. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the facility, and that the associated information is well organized and available for review by authorized assessors.

_____ _____

Bradley A. Weichert
Technical Director

Date

_____ _____

Michael G. Winslow
Quality Assurance Officer

Date

Field Chemist Comments

Comments:

Signature: _____ Date: _____
Title: Field Chemist

Data Review Checklist

_____ Check Data Folder Table of Contents Checklist

_____ Check COCs vs. field data report
- _____ Make sure all sample IDs match and are accounted for.

_____ Data numbers vs field report
- _____ Go through raw data page by page and compare numbers
- _____ Highlight any numbers that need to be changed on the field report

_____ Cover letter

_____ Project narrative
- _____ Change header box and review for accuracy (i.e. water description if waters are run)
- _____ Change date at bottom

_____ Run sequence/surrogate table
- _____ Change header box
- _____ Use daily sequence summaries to get sequence of samples
- _____ Go through raw data to get surrogate recoveries
- _____ Change date at bottom

_____ Matrix spike table
- _____ Change header box
- _____ Comment section
- _____ Change date at bottom
- _____ Review recovery values

_____ Final data report
- _____ Significant figures (only two SF for dilutions)
- _____ Put units on the table

_____ Data report narrative
- _____ Use highlighted preliminary field report
- _____ If significant changes are necessary, inform Director of Operations

_____ COCs
- _____ If yellow pages are gone, client has them from the field; send photocopies of white pages.
- _____ If yellows are still attached, send white pages and keep yellows as originals.

Signature: _____ Date: _____
Title: Data Specialist

Data Folder Table of Contents Checklist

_____ 1. Disk with preliminary field report, FDEP report (if required)

_____ 2. Work Order Form

_____ 3. Field Logs

_____ 4. COCs

_____ 5. Preliminary field data report hardcopy

_____ 6. Comment page (if required) detailing departures from method, problems, etc.

_____ 7. Spike recovery summaries

_____ 8. Initial calibration summaries (if performed)

_____ 9. Tune record (MS only)

_____10. Daily sequence summaries (any pertinent comments should be recorded here including reruns, dilutions, poor recoveries, data not used (incl. 'why'), etc.

_____11. Analysis chromatograms and quantitation reports, in chronological order

_____12. Screening chromatograms

Comments:
_____
_____
_____

Signature: _____Date: _____
Title: Data Review Specialist

Annual System Audit Form

Date: _____ Auditor: _____

Mobile Lab No: ____ Analysis Requested: _____

Sample No.
Project Name:
Client Name:
Date Received:

	Yes	No
Was Chain of Custody properly filled in and signed?		
Does sample receipt logbook match the Chain of Custody?		
Were correct sample fractions received?		
Was the proper method chosen?		
Are bench sheets available for the analysis?		
Was analysis performed within holding times?		
Are all data calculations correct?		
Is there QC with the sample analysis batch?		
Was the project file reviewed?		
Does the instrument log date match the bench sheet date?		
Does the bench data match the data on the report?		
Were there any deficiencies noted?		
Are dilution factors documented?		
Is a run log included and complete?		
Is standard prep log complete?		

Comments	
Corrective Action	

APPENDIX C

Final Report

KB LABS, INC.
6821 Southwest Archer Road
Gainesville, Florida 32608

telephone (352) 367-0073
fax (352) 367-0074

June 1, 2001

Paul Bunyan
Big Boy Environmental, Inc.
12345 Tall Guy Blvd
Oxtown, FL 33333

Re: Final Analytical Report, Big Ugly, Oxtown, FL

Dear Mr. Bunyan:

Enclosed is the final report of the on-site analysis performed by KB Labs, Inc. at the Big Ugly site in Oxtown, FL. On-site analyses were performed May 23 – May 24, 2001. Included are a brief project narrative, tables listing quality control results, final analytical results, and sample chain-of-custody form. This information will also be sent electronically. Including this cover page, the Final Report includes eight pages.

KB Labs' mobile laboratories have been inspected by the FDOH Bureau of Laboratories and have been recommended for NELAP Certification as of April 1, 2003. Our personnel, methodology, proficiency testing, and quality assurance requirements complied with the guidelines of Chapter 64E-1 of the Florida Administrative Code and with the consensus standards adopted at the National Environmental Laboratory Accreditation Conference (NELAC). Data for the site referenced above were determined in accordance with published procedures under Test Methods for Evaluating Solid Waste (EPA SW-846, Update III Revised May 1997). Unless otherwise indicated on the quality control narrative accompanying the data report, the quality assurance and quality control procedures performed in conjunction with analysis of groundwater samples demonstrated that the reported data met our standards for accuracy and precision under NELAC Standards.

If you have any questions, please do not hesitate to call me or Kelly Bergdoll, President of KB Labs, at (352) 367-0073.

Sincerely,

KB Labs, Inc.

Todd Romero
Director of Operations

KB LABS, INC.

PROJECT NARRATIVE

Client: Big Boy Environmental, Inc.	**Driller/Sampler:** Big Rig, Inc.	**Analyst:** M. Mathews
Site: Big Ugly, Oxtown, FL	**KB Project Manager:** Kelly Bergdoll	**KB Project No.** 0333
Onsite Dates: 5/23/01 – 5/24/01	**Client Project Manager:** Paul Bunyan	**Matrix:** Water

Project Scope

On May 23 – May 24, 2001, a total of eight (8) water samples were collected at the Big Ugly site in Oxtown, FL. Samples were analyzed onsite in the KB Labs mobile facility. The samples were analyzed for benzene, toluene, ethylbenzene, m&p-xylene, o-xylene, MTBE, naphthalene, and Diesel Range Organics (DRO).

Analytical Procedure

VOCs – All water samples were analyzed using SW846 Method 5030/8260 for waters. Ten (10) milliliters (mL) of water were purged with helium and the volatile organic compounds (VOCs) were collected on a solid-phase adsorption trap. The adsorption trap was heated and back-purged with helium and the components were separated by capillary column gas chromatography and measured with a mass spectrometer (GC/MS) operated in the electron impact full-scan mode. The individual VOCs in the samples were measured against corresponding VOC standards.

DRO – All water samples were first extracted in a vacuum extraction manifold using Sep-Pak C18 cartridges (2-gram). Sample volumes varied (20 – 200 mL) depending upon particulate content. The C18 cartridges were then solvent extracted with 5 mL of hexane. Samples extracts were then analyzed by gas chromatography/flame ionization detector (GC/FID). DRO in the samples was then measured against a corresponding diesel standard.

Analytical Results

Laboratory results were provided to the client on an as-completed or next-day basis. Final results of the on-site analyses are provided in a standard Excel spreadsheet format. The data produced and reported in the field has been reviewed and approved for this final report by the KB Labs Quality Assurance (QA) Officer.

Quality Control (QC) Data

Surrogate Recoveries (VOCs only) – Tables 1.1 – 1.2 list the daily analytical sequence and percent recovery results for surrogate compounds which were added to all analyses. Four (4) surrogate compounds were added to each analysis in order to continually monitor general method performance.

Matrix Spike and Laboratory Control Spike Recoveries – Table 2 lists the percent recovery results for matrix spike samples and /or laboratory control samples. A known amount of selected target compounds was added to selected field samples and/or to a laboratory blank sample in order to monitor the performance of the compounds in the actual matrix and in the laboratory blank sample.

Method Blanks – Daily analysis of laboratory reagent water samples was performed in order to monitor the cleanliness of the analytical system. No target compounds were detected on or above the reporting limits.

Signature: _____

Title: Director of Operations Date: June 1, 2001

KB LABS, INC.
6821 Southwest Archer Road
Gainesville, Florida 32608

telephone (352) 367-0073
fax (352) 367-0074

Table 1-1: VOC Analysis Sequence/Surrogate Percent Recoveries (5/23/01)

Client: Big Boy Environmental, Inc.	**Driller/Sampler**: Big Rig, Inc.	**Analyst**: M. Mathews
Site: Big Ugly, Oxtown, FL	**KB Labs Project Manager**: Kelly Bergdoll	**KB Labs Project No:** 0333
On-site Dates: 5/23/01 - 5/24/01	**Client Project Manager**: Paul Bunyan	**Matrix:** Water

Station/Sample ID	*Control Limits>>*	S1* *(80-120)*	S2* *(80 - 120)*	S3* *(80 - 120)*	S4* *(80 - 120)*	
CCS 20 ug/L		81	98	97	89	
Method Blank		82	97	95	89	
SMP1		97	96	93	83	
SMP2		85	106	93	82	
SMP3		88	79	95	84	S2 low
SMP4		91	100	96	86	
SMP3 MS		90	94	93	84	
SMP3 MSD		86	95	93	85	
CCS 20 ug/L		85	100	94	89	

* Surrogate Compounds
S1 = 1,2-Dichloroethane-D4
S2 = 1,4-Difluorobenzene
S3 = Toluene - D8
S4 = 4 - Bromofluorobenzene

Signature:_____

Title: Data Specialilst

Date: June 1, 2001

Table 1-2: VOC Analysis Sequence/Surrogate Percent Recoveries (5/24/01)

Client: Big Boy Environmental, Inc.	**Driller/Sampler**: Big Rig, Inc.	**Analyst**: M. Mathews
Site: Big Ugly, Oxtown, FL	**KB Labs Project Manager**: Kelly Bergdoll	**KB Labs Project No:** 0333
On-site Dates: 5/23/01 - 5/24/01	**Client Project Manager**: Paul Bunyan	**Matrix:** Water

	Control	**S1***	**S2***	**S3***	**S4***	
Station/Sample ID	***Limits>>***	***(80-120)***	***(80 - 120)***	***(80 - 120)***	***(80 - 120)***	
CCS 20 ug/L		91	101	92	87	
Method Blank		79	95	96	88	S1 low
REF/LCS		121	103	94	84	S1 high
SMP5		80	96	91	83	
SMP6		120	115	95	93	
SMP7		83	93	97	89	
SMP8		80	95	98	88	
CCS 20 ug/L		119	100	90	82	

* Surrogate Compounds

S1 = 1,2-Dichloroethane-D4

S2 = 1,4-Difluorobenzene

S3 = Toluene - D8

S4 = 4 - Bromofluorobenzene

Signature: _____

Title: Data Specialist

Date: June 1, 2001

Table 2: VOC Spike Compound Percent Recoveries

Client: Big Boy Environmental	**Driller/Sampler**: Big Rig, Inc.	**Analyst**: M. Mathews
Site: Big Ugly, Oxtown, FL	**KB Labs Project Manager**: Kelly Bergdoll	**KB Labs Project No:** 0333
On-site Dates: 5/23/01 - 5/24/01	**Client Project Manager**: Paul Bunyan	**Matrix:** Water

Spike Compounds * > >	**VOC1**	**VOC2**	**VOC3**	**VOC4**	**VOC5**	**VOC6**	**VOC7**	**VOC8**			
Control Limits ** > >	*70-130*	*75-119*	*79-114*	*81-114*	*74-120*	*78-116*	*70-130*	*70-130*		**Comment**	
Warning Limits ** > >	*80-120*	*82-111*	*85-108*	*86-108*	*82-113*	*85-110*	*80-120*	*80-120*			
RPD Limit > >	*20*	*20*	*20*	*20*	*20*	*20*	*20*	*20*			
Station/Sample ID:											
SMP3 MS	77	91	98	94	92	93	100	97			
SMP3 MSD	85	92	99	94	91	93	116	91			
RPD	10	1	1	0	1	0	15	6			
REF/LCS	NA	95	97	92	96	99	120	111			

** Control limits based historical matrix spike recoveries.

* Spike Compounds

VOC1 = MTBE
VOC2 = Benzene
VOC3 = Toluene
VOC4 = Ethylbenzene
VOC5 = m&p-Xylene
VOC6 = o-Xylene
VOC7 = Naphthalene
VOC8 = Diesel

Signature: _____

Title: Data Specialist

Date: June 1, 2001

Analytical Data
Big Ugly, Oxtown, FL
5/23/01 – 5/24/01

	Reporting Limits (ug/L)	SMP1	SMP2	SMP3	SMP4	SMP5	SMP6	SMP7	SMP8				
MTBE	<1	<1	<1	<1	<1	<1	<1	<1	<1				
Benzene	<1	<1	<1	<1	<1	<1	<1	<1	<1				
Toluene	<1	<1	<1	<1	<1	<1	<1	<1	<1				
Ethylbenzene	<1	<1	<1	<1	<1	<1	<1	<1	<1				
m&p-Xylene	<1	<1	1.5	<1	<1	<1	<1	<1	<1				
o-Xylene	<1	<1	<1	<1	<1	<1	<1	<1	<1				
Naphthalene	<1	<1	<1	<1	1.7	<1	<1	2.8	<1				
DRO (mg/L)	See Note	<2.0	<2.9	<2.0	<4.3	<2.0	<4.0	<4.3	<5.0				

Note: DRO reporting limits vary with the total volume of sample processed. This volume will vary depending the particulate content of the sample.

Signature:_____

Title: Data Specialist

Date: 6/1/01

FLORIDA DEPARTMENT OF HEALTH

State of Florida
Department of Health, Bureau of Laboratories

This is to certify that

E82816

K B LABS, INC.; KB-3
25132 SW 1ST AVENUE AMERICA'S BODY COMPANY S/N 10959
(MOBILE LAB)
NEWBERRY, FL 32669

has complied with Florida Administrative Code 64E-1,
for the examination of Environmental samples in the following categories

NON-POTABLE WATER - VOLATILE ORGANICS, SOLID AND CHEMICAL MATERIALS - VOLATILE ORGANICS

Continued certification is contingent upon successful on-going compliance with the NELAC Standards and FAC Rule 64E-1 regulations. Specific methods and analytes certified are cited on the Laboratory Scope of Accreditation for this laboratory and are on file at the Bureau of Laboratories, P. O. Box 210, Jacksonville, Florida 32231. Clients and customers are urged to verify with this agency the laboratory's certification status in Florida for particular methods and analytes.

EFFECTIVE July 01, 2009 THROUGH June 30, 2010

Max Salfinger, M.D.
Chief, Bureau of Laboratories
Florida Department of Health
DH Form 1697, 7/04
NON-TRANSFERABLE E82816-12-07/01/2009
Supersedes all previously issued certificates

Laboratory Scope of Accreditation

Attachment to Certificate #: E82816-12, expiration date June 30, 2010. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E82816 **EPA Lab Code: FL01171** **(352) 367-0073**

E82816
K B Labs, Inc.; KB-3
25132 SW 1st Avenue
America's Body Company S/N 10959 (mobile lab)
Newberry, FL 32669

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,1,1-Trichloroethane	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,1,2,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,1,2-Trichloroethane	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,1-Dichloroethane	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,1-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,1-Dichloropropene	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,2,3-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,2,3-Trichloropropane	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,2,4-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,2,4-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,2-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,2-Dichloroethane	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,3,5-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,3-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,3-Dichloropropane	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,4-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
2,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	2/7/2008
2-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	2/7/2008
4-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	2/7/2008
Benzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
Bromobenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
Bromodichloromethane	EPA 8260	Volatile Organics	NELAP	2/7/2008
Bromoform	EPA 8260	Volatile Organics	NELAP	2/7/2008
Carbon tetrachloride	EPA 8260	Volatile Organics	NELAP	2/7/2008
Chlorobenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
Chloroethane	EPA 8260	Volatile Organics	NELAP	2/7/2008
Chloroform	EPA 8260	Volatile Organics	NELAP	2/7/2008
cis-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	2/7/2008
cis-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	2/7/2008
Dibromochloromethane	EPA 8260	Volatile Organics	NELAP	2/7/2008
Dibromomethane	EPA 8260	Volatile Organics	NELAP	2/7/2008

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Newberry, FL 32669

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Dichlorodifluoromethane	EPA 8260	Volatile Organics	NELAP	2/7/2008
Ethylbenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
Isopropylbenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
Methyl bromide (Bromomethane)	EPA 8260	Volatile Organics	NELAP	2/7/2008
Methyl chloride (Chloromethane)	EPA 8260	Volatile Organics	NELAP	2/7/2008
Methyl tert-butyl ether (MTBE)	EPA 8260	Volatile Organics	NELAP	2/7/2008
Methylene chloride	EPA 8260	Volatile Organics	NELAP	2/7/2008
n-Butylbenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
n-Propylbenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
p-Isopropyltoluene	EPA 8260	Volatile Organics	NELAP	2/7/2008
sec-Butylbenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
Styrene	EPA 8260	Volatile Organics	NELAP	2/7/2008
tert-Butylbenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
Tetrachloroethylene (Perchloroethylene)	EPA 8260	Volatile Organics	NELAP	2/7/2008
Toluene	EPA 8260	Volatile Organics	NELAP	2/7/2008
trans-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	2/7/2008
trans-1,3-Dichloropropylene	EPA 8260	Volatile Organics	NELAP	2/7/2008
Trichloroethene (Trichloroethylene)	EPA 8260	Volatile Organics	NELAP	2/7/2008
Trichlorofluoromethane	EPA 8260	Volatile Organics	NELAP	2/7/2008
Vinyl chloride	EPA 8260	Volatile Organics	NELAP	2/7/2008
Xylene (total)	EPA 8260	Volatile Organics	NELAP	2/7/2008

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E82816
K B Labs, Inc.; KB-3
25132 SW 1st Avenue
America's Body Company S/N 10959 (mobile lab)
Newberry, FL 32669

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,1,1-Trichloroethane	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,1,2,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,1,2-Trichloroethane	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,1-Dichloroethane	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,1-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,1-Dichloropropene	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,2,3-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,2,3-Trichloropropane	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,2,4-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,2,4-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,2-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,2-Dichloroethane	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,3,5-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,3-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,3-Dichloropropane	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,4-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
2,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	2/7/2008
2-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	2/7/2008
4-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	2/7/2008
Benzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
Bromobenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
Bromodichloromethane	EPA 8260	Volatile Organics	NELAP	2/7/2008
Bromoform	EPA 8260	Volatile Organics	NELAP	2/7/2008
Carbon tetrachloride	EPA 8260	Volatile Organics	NELAP	2/7/2008
Chlorobenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
Chloroethane	EPA 8260	Volatile Organics	NELAP	2/7/2008
Chloroform	EPA 8260	Volatile Organics	NELAP	2/7/2008
cis-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	2/7/2008
cis-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	2/7/2008
Dibromochloromethane	EPA 8260	Volatile Organics	NELAP	2/7/2008
Dibromomethane	EPA 8260	Volatile Organics	NELAP	2/7/2008

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Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Dichlorodifluoromethane	EPA 8260	Volatile Organics	NELAP	2/7/2008
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Isopropylbenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
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Methyl chloride (Chloromethane)	EPA 8260	Volatile Organics	NELAP	2/7/2008
Methyl tert-butyl ether (MTBE)	EPA 8260	Volatile Organics	NELAP	2/7/2008
Methylene chloride	EPA 8260	Volatile Organics	NELAP	2/7/2008
n-Butylbenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
n-Propylbenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
p-Isopropyltoluene	EPA 8260	Volatile Organics	NELAP	2/7/2008
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trans-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	2/7/2008
trans-1,3-Dichloropropylene	EPA 8260	Volatile Organics	NELAP	2/7/2008
Trichloroethene (Trichloroethylene)	EPA 8260	Volatile Organics	NELAP	2/7/2008
Trichlorofluoromethane	EPA 8260	Volatile Organics	NELAP	2/7/2008
Vinyl chloride	EPA 8260	Volatile Organics	NELAP	2/7/2008
Xylene (total)	EPA 8260	Volatile Organics	NELAP	2/7/2008